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Application of poly(butyl methacrylate-co-ethylene glycol dimethacrylate) monolith microextraction coupled with high performance liquid chromatography to the determination of polycyclic aromatic hydrocarbons in smoked meat products

Wei Liu [a], Jurui Qi [a], Linna Yan [b], Qiong Jia [a,*], Cong Yu [c]

[a] College of Chemistry, Jilin University, Changchun 130022, PR China
[b] College of Biological and Agricultural Engineering, Jilin University, Changchun 130022, PR China
[c] State Key Laboratory of Electroanalytical Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, PR China

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ABSTRACT

This paper presents a study of the synthesis of a polymer monolith column and its application to the analysis of PAHs in smoked meat products. A poly(butyl methacrylate-co-ethylene glycol dimethacrylate) monolith capillary has been successfully prepared with in situ polymerization method. The polymer monolith microextraction combined with HPLC determinations is employed for the analysis of naphthalene, biphenyl, phenanthrene, and anthracene. Various parameters affecting the extraction efficiency have been investigated and optimized. Under the optimum experimental conditions, the method provides an acceptable linearity (2–10,000 μg/L), low limits of detection (1.4–2.0 μg/L), and good precision (intraday relative standard deviations < 4.1%, interday relative standard deviations < 5.7%). When applied to the determination of the four PAHs in smoked meat samples, recoveries are obtained in the range of 86.6–101.5%.

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1. Introduction

As a group of organic compounds consisting two or more condensed aromatic rings, polycyclic aromatic hydrocarbons (PAHs) are considered to be top of the list of the most hazardous substances [1]. They have been included in European Union (EU) and Environmental Protection Agency (EPA) priority pollutant lists. Therefore, it is of great importance for the identification and determination of PAHs. Because of the low level concentration and complex matrix effect, the pretreatment and concentration step is inevitable before detection of PAHs to enhance the sensitivity and selectivity. As traditional techniques, liquid–liquid extraction (LLE) [2,3] and solid phase extraction (SPE) [4–6] have been commonly used for the preconcentration of PAHs. As is well known, LLE and SPE have the disadvantages of being tedious, time-consuming, or requiring toxic organic solvents. Therefore, many other separation techniques have been developed. Accelerated or pressurized extraction [7–9], cloud-point extraction [10,11], membrane-assisted solvent extraction [12], ultrasonic-assisted extraction [13,14], Soxhlet extraction [8,15], microwave-assisted extraction [8,14,16], supercritical fluid extraction [17], and liquid–liquid microextraction [4] have found their applications in the preconcentration of PAHs.

Solid-phase microextraction (SPME), firstly introduced by Arthur and Pawliszyn [18], has been successfully applied to the extraction of various compounds in environmental, industrial, pharmaceutical, and clinical samples. In the SPME technique, the most important problem is to select an appropriate fiber for the extraction of analytes. However, SPME fiber is fragile and has limited lifetime, and the sample carry-over is also a problem. In addition, only several SPME fibers are commercialized, which limits its applications. In-tube SPME [19], using an open tubular fused-silica capillary column with an inner surface coating as the SPME device, is simple and can be easily coupled on-line to HPLC and LC–MS. In-tube SPME allows convenient automation of the extraction process, which not only reduces the analysis time, but also provides better accuracy, precision, and sensitivity than manual off-line techniques.

Various materials have been reported as the extraction media in an in-tube SPME device, e.g. cyanopropyl phenyl methyl-silicone [19]. Monolithic material is a type of separation medium with high speed, high efficiency and high throughput, and has been widely applied as the stationary phase in various chromatographic

* Corresponding author at: College of Chemistry, Jilin University, Renmin Street 5988#, Changchun 130022, PR China. Tel.: +86 431 85095621; fax: +86 431 85095621.
E-mail address: jiaqiong@jlu.edu.cn (Q. Jia).

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methods [20]. Among monolithic materials, organic polymer materials can be easily in situ synthesized by thermal or irradiation initiating the polymerization of certain mixture of monomer, crosslinker, and proper porogenic solvent in a "mold" [21]. The procedure for synthesizing such kind of material is simple and reproducible and they can be easily situated in the separation support, making them an attractive alternative choice for the application as separation media.

As a branch of in-tube SPME, polymer monolith microextraction (PMME), first introduced by Feng's group [22], shows attractive features of being free moving and requiring simple instrument and manipulation. PMME has been applied to the preconcentration step when combined with CE [22,23], ICP-MS [24], HPLC [25–28], HPLC–MS [29,30], GC–MS [31], and MALDI/MS [32]. In our previous work, poly(methacrylic acid-co-ethylene glycol dimethacrylate) monolith microextraction has been coupled to HPLC determinations. The method is proved to be simple, fast, and sensitive when used for monitoring nitroanilines in hair dye samples [25] or phthalate esters in cosmetic products [26].

So far, only a few kinds of organic monolithic materials have been employed in PMME, including poly(methacrylic acid-co-ethylene glycol dimethacrylate) monolith [22,23,25–27,29,31], hydroxyapatite nanoparticles incorporated poly(2-hydroxyethyl methacrylate-co-ethylene dimethacrylate) monolith [32], hydroxylated or gold nanoparticles bonded poly(glycidyl methacrylate-co-ethylene dimethacrylate) monolith [28,30], poly(acrylamide-vinylpyridine-*N*,*N*'-methylene bisacrylamide) monolith [24], and poly(vinyl phenylboronic acid-ethylene glycol dimethacrylate) monolith [33]. Polymethacrylate-based monoliths are conceived as one of the most important types of organic polymer monoliths. As a methacrylate containing 4C atoms in the carbon chain, butyl methacrylate (BMA) has been employed as the monomer to prepare monolith material and applied to the separation of microcystins in capillary-HPLC. The feasibility of BMA-based polymeric monolithic CEC technique on real food, pharmaceutical, and cosmetic product analyses has been evaluated [34]. However, our survey through literature has not applied to poly(BMA)-based monoliths in the field of PMME.

Smoked meat products present a significant part of the human diet because of their good taste, high nutritional value, and large variety of products. However, it is well known that hundreds of individual PAHs may be formed and released during the incomplete combustion or thermal decomposition of the organic material. Therefore, monitoring of PAHs levels in food samples is important in evaluating the risks associated with human consumption of meat products. The aim of the present study is to develop a sensitive and simple method for the determination of PAHs combining PMME with HPLC. A poly(BMA-co-ethylene glycol dimethacrylate) monolith capillary has been synthesized. Naphthalene, biphenyl, phenanthrene, and anthracene are chosen as respensitives. The PMME–HPLC method is optimized and applied to the preconcentration of PAHs. Finally, the optimized method has been validated and applied to the determination of the target analytes in different smoked meat products.

2. Materials and methods

2.1. Chemicals and materials

BMA, 2,2′-azobis(2-methylpropionitrile) (AIBN), 1,4-butanediol, and 1-butanol, were obtained from Guangfu Research Institute of fine chemicals (Tianjin, China). Ethylene glycol dimethacrylate (EGDMA), 3-(trimethoxysilyl)propylmethacrylate, naphthalene, biphenyl, phenanthrene, and anthracene were purchased from Aladdin Reagent Co., Ltd., China. The stock standard solutions of 100 μg/mL of PAHs were prepared in methanol (HPLC grade, Merck, Germany). The standard working solutions were daily prepared by appropriate dilution from the individual stocks. The stock solutions and diluted standard solutions were stored in glass volumetric flasks in the dark at 4 °C. All solvents and solutions for HPLC analysis were filtered through a Millipore filter (pore size 0.45 μm). All the glassware used in the study was previously washed with acetone, *n*-hexane, and dichloromethane and finally dried in an oven at 250 °C. Double distilled water (DDW) was used for all experiments.

2.2. Instrument and analytical conditions

Chromatographic analysis was performed on an Agilent 1100 liquid chromatography system, equipped with a quaternary pump and degasser, a thermostated autosampler (4 °C) and column compartment (35 °C), a multiple wavelength detector, and ChemStation software. A reverse phase Agilent Zorbax Eclipse XDB-C_{18} column (4.6 × 250 mm, 5 μm) was used for the chromatographic separation. A phenomenex C_{18} security guard column (4.0 × 3.0 mm) from Phenomenex, Torrance, Canada was used to protect the column. Methanol–H_2O (85:15, v/v) was employed as the mobile phase with a flow rate of 1.0 mL/min. The injection volume was 5 μL and the column temperature was 30 °C. The preferential detection wavelength was 250 nm.

For pH measurements, a pHS-3C digital pH meter (Shanghai Rex Instruments Factory, China) was employed. The Milli-Q SP system (Millipore, Milford, MA, USA) was used to prepare deionized water. An LSP01-1A programmable syringe pump (Baoding Longer Precision Pump Co., Ltd., Hebei, China) was used for passing through solutions. Smoked meat samples were cut into pieces by a JYS-A801 mixer (Guangdong Joyoung Co., Ltd., China). Cell disruption experiments of meat samples were carried out by a JYD-150 ultrasonic cell crusher (Shanghai Zhi Sun Instrument Co., Ltd., China). The rotary evaporator with a model of Laborota 4010 Digital was from Heidolph, Germany.

2.3. Preparation of poly(BMA–EGDMA) monolithic capillary column

The fused silica capillary (20 cm × 0.32 mm, i.d., Hebei Yongnian Fiber Plant, China) was washed subsequently with acetone, 0.1 mol/L NaOH, H_2O, 0.1 mol/L HCl, H_2O, and acetone for 10 min. Before polymerization, the inner wall of the fused silica capillary was modified with 3-(trimethoxysilyl)propylmethacrylate (30%, v/v in acetone) to improve the adhesion of the monolith bed to the capillary wall. After sealing the capillary two ends with silicon rubber, the reaction was allowed to perform at 40 °C for 14 h. The capillary was then washed with acetone thoroughly and purged with N_2 for 2 h.

In order to perform the polymerization step, AIBN was dissolved in mixtures of BMA and EGDMA (BMA:EGDMA:AIBN = 44.5:54.5:1, w/w) to form solution A. Ternary porogen solvent (B), containing 1-butanol, 1,4-butanediol, and H_2O (1-butanol:1,4-butanediol:H_2O = 60:30:10, w/w) was slowly added into A with a ratio of 40:60 (A:B, v/v). The mixtures of A and B were filled in the activated capillary and then sealed with silicon rubber at both ends immediately. After reacting at 60 °C for 20 h, the capillary was washed with methanol to remove the untreated component and porogenic solvent [34,35].

The PMME device was prepared according to Zhang et al.'s work [22]. Briefly, a syringe barrel was coupled seamlessly to one end of the pinhead of the syringe, while on the other end of the pinhead, its metallic needle was replaced by a 2 cm long part cut from the

prepared monolithic capillary, the outside wall of which was coated uniformly with adhesive.

2.4. Poly(BMA–EGDMA) monolith microextraction procedure

The PMME procedure consisted of four successive steps, preconditioning, sample loading, washing, and desorption [26]. For preconditioning, 0.2 mL methanol was introduced into the syringe and pushed to pass through the monolithic capillary at a speed of 0.05 mL/min, and then 0.5 mL NaH_2PO_4 (pH 4.5) was expelled at a flow rate of 0.15 mL/min. After that, 1.5 mL sample solution was ejected at 0.1 mL/min in the same way. In the washing step, 0.2 mL NaH_2PO_4 (pH 4.5) was expelled to flow through the capillary at a flow rate of 0.15 mL/min. In order to avoid the contamination of the eluate, the residual solution in the pinhead and the monolithic capillary was removed with a clean syringe. In the desorption step, a mixture of methanol and H_2O (85:15, v/v) was injected to the monolithic capillary at 0.05 mL/min for 1 min and the eluate was collected into a vial for HPLC determinations.

2.5. Sample preparation

Before use, all smoked meat samples were stored in their original packaging under the recommended conditions. Firstly, the spiked or blank samples were cut into pieces and homogenized to powder. After dissolved with acetonitrile (sample:acetonitrile = 1:3, w/v), the samples were extracted by an ultrasonic cell crusher. The supernatants were concentrated to a small volume using a rotary evaporator at 50 °C [36,37]. Finally, the sample was reconstituted to 1 mL with a buffer solution of NaH_2PO_4 (pH 4.5), and after filtered with a membrane filter (0.45 μm), prepared for the PMME steps.

3. Results and discussion

3.1. Effects of experimental conditions on the extraction efficiency

The extraction has been optimized with 1.0 μg/mL naphthalene, biphenyl, phenanthrene, and anthracene. Several experimental parameters affecting the extraction efficiency such as sample volume, sample flow rate, and eluent flow rate have been investigated.

The effect of sample volume on the extraction efficiency has been examined by ejecting different volumes of sample solutions containing 1.0 μg/mL of each target analyte through the monolithic capillary when other experimental conditions are kept constant. As shown in Fig. 1, the peak area increases with increasing sample volume within the range of 0.5–3.0 mL (corresponding to 5–30 min when the sample flow rate is fixed at 0.1 mL/min). The extraction equilibrium has not been reached even when 3.0 mL of sample solution is fed, which indicates that the poly(BMA–EGDMA) monolithic capillary has remarkable extraction capacity for the target PAHs. A lower limit of detection can be expected to be achieved by the monolithic capillary when compared with direct injection. Considering the analytical speed, a sample volume of 1.5 mL has been selected as a compromise.

When the concentration and sample volume of PAHs have been fixed, the effect of sample flow rate has been investigated. Sample flow rate is one of the important experimental parameters which may not only affect the recoveries of the target analytes, but also control the time of analysis. Fig. 2 indicates the effect of sample flow rate on the extraction efficiency in the range of 0.05–0.3 mL/min. It can be seen that the extraction efficiency firstly increases with increasing sample flow rates and then reaches maximum at 0.1 mL/min. The reason may be that too slow flow rate is not beneficial to mass transfer during the extraction, while too fast flow rate may cause less contact time between the analytes and

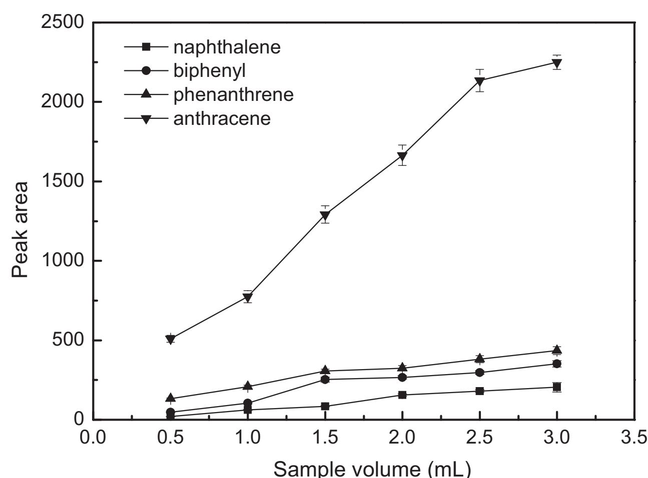

Fig. 1. Effect of sample volume on the extraction of PAHs on polymer monolith. Concentration of the target analyte: 1.0 μg/mL; sample flow rate: 0.1 mL/min; eluent flow rate: 0.05 mL/min.

the extractions phase. Finally, a sample flow rate of 0.1 mL/min is chosen as an optimum.

In order to obtain a quantitative recovery of analytes, the choice of a suitable eluent and the optimum of eluent flow rate are very important. In the HPLC determination step, a mixture of methanol and H_2O (85:15, v/v) has been employed as the mobile phase. Therefore, such a mixture is considered as the eluent preferably. In all the experiments, 0.05 mL methanol–H_2O (85:15, v/v) is employed as the eluent. The enrichment factor, calculated by the ratio of the sample volume to the eluent volume, can thus be obtained as 30. In addition, the effect of eluent flow rate on the extraction efficiency has been investigated in the range of 0.025–0.175 mL/min. High flow rate can lead to short experimental time. However, when the flow rate is too high, poor stability of the flow rate may be encountered because of the high column pressure. As can be seen from Fig. 3, a quantitative recovery can be achieved when methanol–H_2O (85:15, v/v) is chosen as the eluent with a flow rate of 0.05 mL/min. Therefore, 0.05 mL/min has been employed as the optimum eluent flow rate in view of the column pressure and the desorption time.

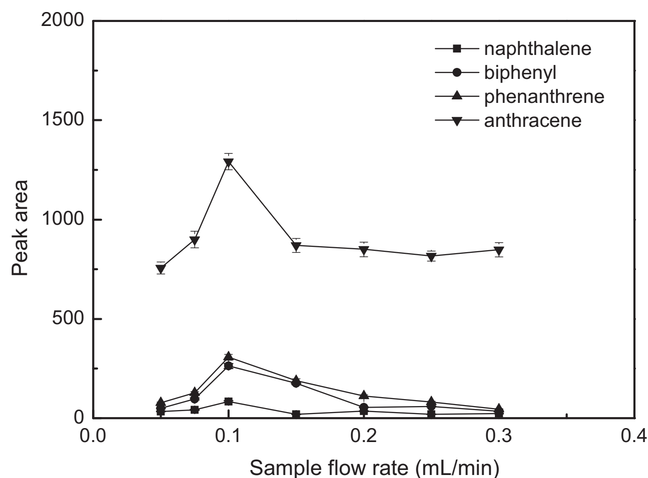

Fig. 2. Effect of sample flow rate on the extraction of PAHs on polymer monolith. Concentration of the target analyte: 1.0 μg/mL; sample volume: 1.5 mL; eluent flow rate: 0.05 mL/min.

Table 1
Linear range, LOD, LOQ, intraday and interday repeatabilities of peak areas for PMME of PAHs.[a]

PAHs	Linear range (μg/L)	r	LOD (μg/L)	LOQ (μg/L)	Intraday repeatability (%)	Interday repeatability (%)
Naphthalene	2–10,000	0.9999	2.0	6.7	3.3	4.6
Biphenyl	2–10,000	0.9999	1.7	5.6	2.6	4.1
Phenanthrene	2–10,000	0.9991	0.8	2.7	3.6	5.7
Anthracene	2–10,000	0.9995	0.4	1.4	4.1	3.9

[a] Repeatabilities (RSD) are for PAHs at a concentration of 1.0 μg/mL (n = 7).

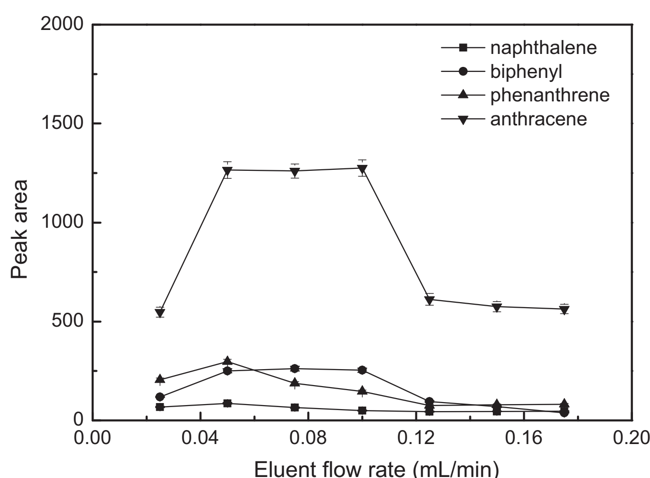

Fig. 3. Effect of eluent flow rate on the extraction of PAHs on polymer monolith. Concentration of the target analyte: 1.0 μg/mL; sample volume: 1.5 mL; sample flow rate: 0.1 mL/min.

3.2. Analytical performance

Under the above optimum experimental conditions, the PMME method has been evaluated by linear range, limit of detection (LOD), limit of quantification (LOQ), and repeatability. Results are shown in Table 1. Linearity has been observed over the range of 2–10,000 μg/L for the four PAHs. Regression coefficients of correlation (r) range from 0.9991 to 0.9999. LOD and LOQ, calculated using the IUPAC recommendation (based on three and ten times the standard deviation of the peak area, respectively) have also been calculated. As shown in Table 1, the method has low LOD and LOQ values.

The reproducibility of the developed method has been evaluated by the intraday and interday precisions. Results are also shown in Table 1. The intraday relative standard deviations (RSDs) are determined in seven replicates for all the studied PAHs. The interday precision data are obtained by analysis of the PAHs in five consecutive days. The intraday and interday RSD values are in the range of 2.6–4.1% and 3.9–5.7%, respectively.

The present PMME–HPLC method has been compared to some preconcentration methods for the extraction of PAHs. The LOD values are comparable to the ones obtained with other methods, such as homogeneous LLE (0.02–0.18 μg/L) [3], dispersive liquid–liquid microextraction and dispersive μ-solid-phase extraction (0.01–0.06 μg/L) [4], SPE (0.001–0.15 μg/L) [5], cloud-point extraction (0.0002–137 μg/L) [11], and microwave-assisted extraction (0.09–1.5 μg/L) [16].

3.3. Stability of poly(BMA–EGDMA) monolithic capillary

The stability of the poly(BMA–EGDMA) monolithic capillary has also been investigated because it is one of the key factors in evaluating the performance of PMME. After each extraction cycle, 0.2 mL methanol is passed through the monolithic capillary in order to eliminate the memory effect. No signals have been detected for a blank injection after the highest standard concentration, implying that no carry-over will affect the next analysis. The extraction efficiency has been verified within a successive 4 months of operation under the experimental conditions. There are no significant changes about the extraction efficiency, indicating the poly(BMA–EGDMA) monolithic capillary is feasible to be used multiply.

3.4. Analysis of real smoked meat samples

The improved method has been applied to analyze real smoked meat samples from local supermarkets. Recovery experiments have

Table 2
Recovery values (%) for smoked meat samples (n = 3).[a]

Samples	PAHs	Found (μg/kg)	Recovery mean (%) ± SD	
			Level1	Level2
Smoked beef	Naphthalene	–	100.8 ± 2.6	99.7 ± 3.6
	Biphenyl	–	89.7 ± 4.2	98.6 ± 4.3
	Phenanthrene	27.8	92.8 ± 2.2	88.5 ± 3.4
	Anthracene	1.6	94.3 ± 1.2	100.7 ± 3.8
Smoked sausage	Naphthalene	–	94.1 ± 4.7	87.8 ± 4.4
	Biphenyl	–	99.4 ± 4.2	91.0 ± 5.1
	Phenanthrene	18.0	99.3 ± 3.2	88.9 ± 3.7
	Anthracene	2.4	91.5 ± 2.5	98.5 ± 4.7
Smoked garlic sausage	Naphthalene	–	90.4 ± 3.9	92.1 ± 2.9
	Biphenyl	2.4	99.8 ± 4.2	101.0 ± 5.2
	Phenanthrene	32.7	92.6 ± 4.8	88.7 ± 2.0
	Anthracene	1.6	101.5 ± 3.8	93.5 ± 3.4
Smoked bacon	Naphthalene	–	94.7 ± 3.1	89.9 ± 2.7
	Biphenyl	–	86.6 ± 4.2	92.5 ± 4.4
	Phenanthrene	34.7	90.5 ± 1.5	87.4 ± 3.5
	Anthracene	4.9	92.0 ± 2.2	93.1 ± 3.6

[a] The concentrations of naphthalene, biphenyl, phenanthrene, and anthracene spiked to smoked meat samples are 81.6, 40.8, 40.8, and 20.4 μg/kg (Level1); 244.9, 122.5, 122.5, and 61.2 μg/kg (Level2), respectively.

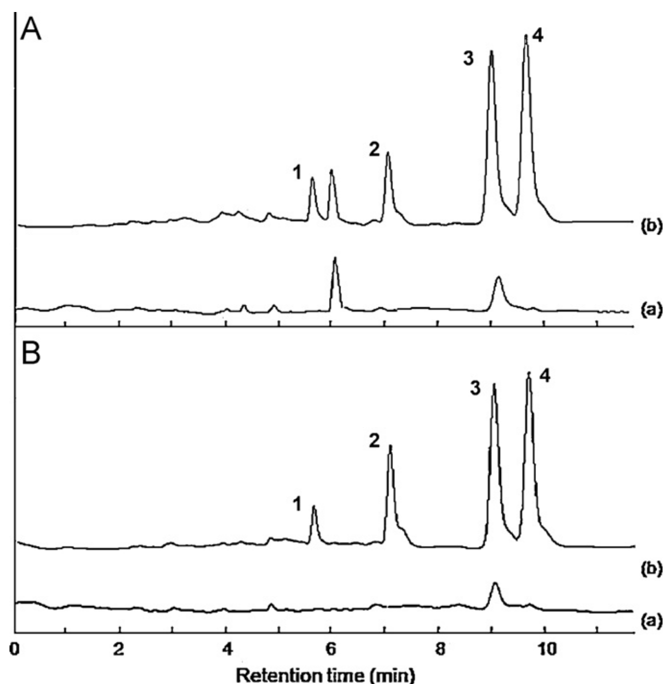

Fig. 4. Chromatograms of smoked meat samples obtained by PMME procedures. (A) Smoked beef; (B) smoked sausage. (a) Blank smoked meat sample, (b) smoked meat sample spiked with 81.6 μg/kg naphthalene, 40.8 μg/kg biphenyl, 40.8 μg/kg phenanthrene, and 20.4 μg/kg anthracene. Peaks: 1-naphthalene, 2-biphenyl, 3-phenanthrene, and 4-anthracene.

been performed in 4 real samples to test the application of the method. The recoveries of PAHs from the smoked meat samples spiked with low and high PAHs concentrations are calculated by comparing the actual amounts of the PAHs added to those obtained by calculation. Satisfactory recoveries can be obtained in the range of 86.6–101.5% (Table 2). Typical chromatograms of blank and spiked samples are shown in Fig. 4. It can be concluded that the method is a reliable technique for the analysis of trace PAHs in complex samples.

4. Conclusions

In this study, a polymer monolithic column has been prepared by in situ polymerization of BMA, EGDMA, AIBN, with 1-butanol, 1,4-butanediol, and H_2O as porogens. The synthesis exhibits good reproducibility and high stability. The poly(BMA–EGDMA) monolith has been applied to the extraction of naphthalene, biphenyl, phenanthrene, and anthracene combined with HPLC-UV determinations. Experimental conditions have been optimized including sample volume (1.5 mL), sample flow rate (0.1 mL/min), and eluent flow rate (0.05 mL/min). The developed PMME–HPLC technique is environmentally friendly, simple, cheap, rapid, and precise for the determination of PAHs. It shows feasibilities for future application to residues determinations and prohibited substances measurements from complex foodstuffs.

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