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Preparation of PDMS-coated microspheres by sol–gel method for sorptive extraction of PAHs

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Abstract

In this paper, a novel SPME mode, PDMS-coated solid glass microspheres (SGMs), were prepared by sol–gel method. Using homemade thermal desorption unit coupled with CGC-FID, six PAHs as model analytes, the performance of the new mode was characterized. The new extractive phase exhibited high thermal stability and satisfactory extraction capability. The detection limits were 0.01–0.045 ng/mL, and the linearity was from 0.5 ng/mL to 96 ng/mL. The R.S.D.s of repeatability for retention time and peak area were all within 0.074% and 6.7%, respectively. The recoveries of the PAHs were 78–127% from the samples taken from river water.

Keywords: Sorptive extraction; PDMS-coated microspheres; Sol–gel; PAHs

The solid-phase microextraction (SPME), introduced in 1989 [1], is a solvent-free sample pretreatment technique, which allows extraction, concentration and sample introduction to be done in a single step. So far the SPME has developed into many kinds of modes, such as fiber extraction [1], in-tube sorptive extraction [2], stir bar sorptive extraction (SBSE) [3] and so on [4,5]. In this study, a novel SPME mode, PDMS-coated solid glass microspheres (SGMs), is introduced. Here, the microspheres, about 0.5 mm in diameter, covered with PDMS, are used to extract analytes from samples. Compared with conventional SPME modes, SGMs possess both large extraction phase volume and surface area. And as a common knowledge of sorption principle, not only the total amount but also the surface area of the extraction phase is important to extraction efficiency in sorptive extraction. Therefore, the novel extraction mode is expected to have a higher extraction capacity and rate.

1. Experimental

The solid glass microspheres were washed in turn with distilled water, methylene chloride, 1 mol L^{-1} NaOH and 0.1 mol L^{-1} HCl [6]. Then the microspheres were dried at 120 °C under a flowing N_2 atmosphere for 1 h. Finally, some of them (≈0.2900 g) were immersed into a sol solution consisting of 150 mg OH-PDMS, 300 μL CH_2Cl_2, 40 μL MTMOS, 30 μL PMHs, 50 μL KH-560 and a desired amount of TFA (95%) for 60 min. After that, the coated SGMs

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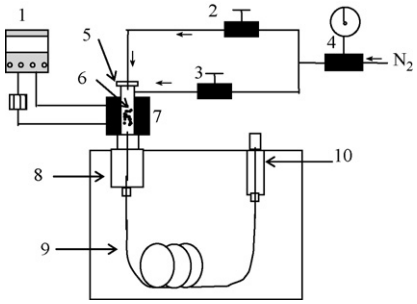

Fig. 1. Schematic diagram of homemade thermal desorption unit coupled with CGC-FID. 1, temperature control apparatus; 2 and 3, flow control valve; 4, pressure regulator; 5, thermal desorption tube; 6, solid glass microspheres; 7, heating block; 8, injector; 9, capillary column; 10, FID detector.

were taken out and exposed to the air for 24 h, followed by gelling the coating in a vacuum desiccator for 10 h. The coated SGMs were purged with N_2 under temperature programming [7], namely heating from 40 °C to 120 °C at 1 °C/min, held for 2 h, and then to 320 °C at 0.5 °C/min, held for 3 h. The amount of PDMS coated on the SGMs can be controlled by the dipping time and times in the sol solution.

When extraction completed, the solution containing SGMs was poured on a piece of stainless-steel net fixed on a beaker, so the SGMs were separated from the solution. After that the SGMs were dried by filter paper, and were placed in a liner in the desorption tube (part 5 in Fig. 1). Then this desorption tube was heated by part 7, and the heating process was controlled by part 1. The analytes released from extraction phase were carried into analysis column by N_2 and focused at the head of the cooling column (35 °C). After a few minutes, the analysis process was started.

2. Results and discussion

The surface of the sol–gel PDMS SGM was checked by SEM, shown in Fig. 2. The coating layer is uniform without cracking, which guaranteed no peeling during extraction. Fig. 3 shows the compare between original and coated microsphere. The amount of PDMS coated on the SGMs is 0.0036 g, being equivalent to 4.0 μL. The thermal stability of the coating on the SGMs was examined by thermal desorption in our homemade thermal desorption unit from

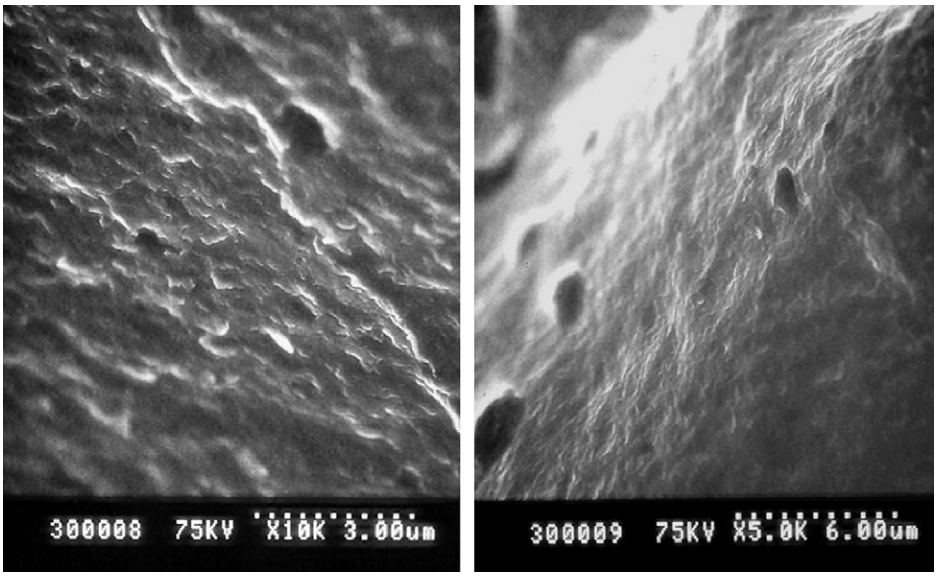

Fig. 2. Scanning electron micrograph of the surface structures of the sol–gel PDMS coating on one SGM, magnification 10,000× and 5000×, 75-kV acceleration.