



Recent developments in modifying polypropylene hollow fibers for sample preparation



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ABSTRACT

Polypropylene hollow fibers (PPHFs) are usually applied for HF liquid-phase microextraction and have proved effective in various applications. By modifying PPHFs with sorbents, they can be used for solid-phase microextraction, and the method is novel, efficient and economical. This article mainly reviews the modification of PPHFs using carbon nanotubes, graphene, ceramic metal oxides and self-synthesized polymers.

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1. Introduction

As a solvent-free sample-pretreatment method, solid-phase microextraction (SPME) was introduced in 1990s by Pawliszyn et al. [1]. Since its discovery, due to the simplicity and low solvent consumption of the method, SPME has been applied to sample preparation [e.g., food [2], environment [3], and plasma [4]]. Also, on-line coupling of SPME with high-performance liquid chromatography (HPLC) [5], gas chromatography (GC) [6] and other techniques [7] has been fully realized.

Although SPME is a potent sample-pretreatment method, SPME fibers have some drawbacks, such as fragility, and stripping of coating [8]. Also, since SPME fibers are relatively expensive, and the prep-

aration procedure is tedious, the fibers have to be used repeatedly, which greatly increases the carry-over effect of this method. As a result, the application of low-cost polypropylene hollow fiber (PPHF) as a way of stabilizing sorbents, instead of fused-silica or steel fibers, is a good alternative.

Due to their low cost, low carry-over effect, low organic-solvent consumption, better sample enrichment and clean-up function, PPHFs are usually used in HF liquid-phase microextraction (HF-LPME) for the protection of microdroplets of extraction solvents, and proved to be practical for the treatment of samples in various areas of study [9,10]. However, compared with the normal SPME, the surface area of HF and the partition coefficient in LPME are relatively small.

Since both normal SPME method and HF-LPME have their disadvantages, to modify HF with different sorbents and to apply in SPME is one way to ameliorate this situation, so, by combining HF with SPME, the carry-over effect of SPME can be reduced and the

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extracted amount in HF-LPME can be increased. In this review, we introduce the development of modifications of HFs with four groups of particular sorbents and their applications in SPME.

2. Carbon-nanotube-reinforced HF membrane

Because of the strong mechanical, electronic and structural properties of carbon nanotubes (CNTs), the application of CNTs in chemical analysis increased rapidly in recent years [11]. By means of theoretical studies, we know that CNTs possess the property of high flux, which is attributed to the smooth surface, frictionless, rapid transport and molecular ordering. Besides being excellent transporters, CNTs are also superior sorbents for various organic and inorganic compounds, because of their large surface area and the hydrophobic characteristics of their surface [12]. Furthermore, CNTs are great candidates for ultratrace accumulation of compounds possessing a π -electron structure, which could adhere to the sp^2 structure of graphene sheets via π - π interactions. Based on these findings, researchers combined CNTs with PPHF to increase the extraction efficiency of HF-LPME and HF-SPME. When employed in SPME, CNTs are usually held in the lumen or immobilized into the pores of the HF and serve as an analyte trap, resulting in higher selectivity and enrichment. In LPME, CNTs are held in the lumen of the HF as the acceptor phase, and their addition could give a strong adsorption capacity. In a new extraction mode, known as solid/liquid phase microextraction, CNTs are held in the pores of the HF, providing an additional avenue for solute transport and acting as effective sorbents. They provide sites at which the analyte molecules can transfer from the donor phase to the acceptor phase (i.e., the analyte molecules can first adsorb on the CNTs, and are then desorbed into the acceptor phase). In this case, the mechanism of solute transfer involves both liquid-phase and solid-phase extractions.

HF-LPME with the mixture of multi-walled CNTs (MWCNTs) and 1-octanol as the acceptor phase has been applied to extract carbamate pesticides in water and fruit samples by Zhao et al. [13]. MWCNTs were ultrasonically well dispersed in 1-octanol. During the extraction process, the mixture was injected into the lumen of the HF and used as the acceptor phase. After the two ends of the HF were sealed, the extraction medium was put into the sample solution for extraction and enrichment. When the extraction was accomplished, the fiber was transferred into methanol for ultrasonic desorption. Finally, a certain amount of desorption solution was injected into HPLC for analysis. The effect of 1-octanol, 1-octanol with MWCNTs, and 1-octanol with oxidized MWCNTs used as acceptor phases were compared. The results showed 1-octanol with MWCNTs obtained the best extraction efficiency. The reason is that both carbamate pesticides and MWCNTs are of weak polarity, so their interactions are strong. The best extraction efficiency was achieved at 1 mg mL⁻¹ of MWCNTs. The clean-up ability of liquid-liquid extraction (LLE), HF-LPME with MWCNTs, and HF-LPME without MWCNTs were compared. LLE resulted in a stronger interfering matrix effect than the other two methods. Compared with HF-LPME without MWCNTs, HF-LPME with MWCNTs had higher enrichment factors (EFs) for the compounds studied, so the proposed method can be a better substitute for the trace analysis of carbamates in water and food samples.

The silica shell of MWCNTs with homogenous thickness was prepared inside the HF by Eshaghi et al. [14]. In this method, the composite of organic and inorganic polymer containing oxidized MWCNTs was prepared by sol-gel technology, in which tetraethyl orthosilicate (TEOS) was used as the precursor and HCl as the acidic catalyst. The liquid sol was injected into fibers for the *in-situ* gelation, then the new SPME adsorbent was prepared. The HF after treatment was directly immersed into the sample for extraction. During desorption, the analyte was rapidly desorbed from the sorbent fiber because of the pore structure of the HF. This HF had no carry-

over effect, since each piece was only used once. It can also be used in complex matrices, as the pore in HF prevents the entrance of large molecules. From the results, it was observed that this method achieved a relatively low limit of detection (LOD) compared with other methods for the determination of phenobarbital in water samples.

Eshaghi et al. modified their previous method [14] and prepared another economical SPME device [15]. The HF was treated with MWCNT-COOH reinforced sol-gel, which was prepared via the reaction of phenyl trimethoxysilane and a basic catalyst, and then both ends of the fiber segment were sealed. 50 mg of MWCNTs in 1 mL of sol solution was selected as the optimized amount for the extraction, and the aging time of 72 min of sol-gel was selected as the optimal. This SPME HF was placed in 25 mL of sample solution for extraction, then the analyte diffused through the membrane into the MWCNTs. The HF was transferred to MeOH for desorption ultrasonically. This method was used to pretreat compounds with medium polarity, such as aflatoxins, and the results showed that this method has high precision and low LODs.

By using sol-gel technology, MWCNTs could be better dispersed inside the HF compared with dispersing MWCNTs with organic solvent. However, MWCNTs were in the lumen of HF for both methods, and when the ultrasonic effect was adopted during desorption, the stability of MWCNTs inside the HF would be questionable.

Accordingly, researchers explored methods for improving the stability of MWCNTs in the HF. Wu et al. filled wall pores of the HF with oxidized MWCNTs in 1-octanol [16], which were injected into the HF by a microsyringe, and then the HF was sonicated until the mixture stuffed the wall pores full, while an inserted stainless-steel wire was used to prevent blockage of the HF. There was an investigation of the effect of different extractants, including HF membranes (HFMs) with 1-octanol alone, with raw MWCNTs and 1-octanol, with oxidized MWCNTs alone and with oxidized MWCNTs and 1-octanol. The results showed that combined use of oxidized MWCNTs and 1-octanol displayed higher extraction efficiency than either alone. Oxidized MWCNTs showed a higher extracted amount than raw MWCNTs, because the surface of oxidized MWCNTs has more oxygenated functional groups, which makes oxidized MWCNTs more hydrophilic, thus more suitable for the extraction of comparatively polar compounds. The established method had comparable results in terms of LODs and relative standard deviations (RSDs) to those of reference methods, but this method is more sensitive and rapid for the determination of triazine herbicides in environmental water and food samples.

Oxidized single-walled carbon nanohorns (o-SWCNHs) were immobilized into the pores of the HF by Jimenez-Soto et al. [17]. First, oxygenated functional groups were introduced to SWCNHs by microwave energy. Second, methanol was added to the vial containing o-SWCNHs, and then the mixture was well dispersed ultrasonically. HF, with an inserted stainless-steel wire to keep the vertical position, was immersed in the mixture. The o-SWCNHs were stuffed homogeneously into the pores or spread on the surface of the HF under sonication. The prepared o-SWCNH-immobilized HF could be used 30 times for SPME without any change in extraction efficiency. The main drawback of this method was that 150 μ L of organic solvent had to be used as desorption solution, while direct injection is usually used in SPME. This method is better than, or comparable to, other analytical methods for the determination of triazine in environmental samples.

The mixture of CNTs and water were pumped into the lumen of the HF under pressure, so that CNTs were stuffed into the wall pores of the HF. This CNT-supported liquid membrane was designed by Hilton et al. for the selective extraction of organic acids [18]. CNTs were first processed by microwaves to be functionalized with -COOH, -NO₂ and -HSO₃, and dispersed in water by sonication. During extraction, the analyte transported from the sample solution to CNTs in the wall pores of the HF, then partitioned to the

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