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Gas chromatographic determination of polycyclic aromatic hydrocarbons in water and smoked rice samples after solid-phase microextraction using multiwalled carbon nanotube loaded hollow fiber

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ABSTRACT

A novel solid-phase microextraction fiber was prepared based on multiwalled carbon nanotubes (MWCNTs) loaded on hollow fiber membrane pores. Stainless steel wire was used as unbreakable support. The major advantages of the proposed fiber are its (a) high reproducibility due to the uniform structure of the hollow fiber membranes, (b) high extraction capacity related to the porous structure of the hollow fiber and outstanding adsorptive characteristics of MWCNTs. The proposed fiber was applied for the microextraction of five representative polycyclic aromatic hydrocarbons (PAHs) from aqueous media (river and hubble–bubble water) and smoked rice samples followed by gas chromatographic determination. Analytical merits of the method, including high correlation coefficients [(0.9963–0.9992) and (0.9982–0.9999)] and low detection limits [(9.0–13.0 ng L^{-1}) and (40.0–150.0 ng kg^{-1})] for water and rice samples, respectively, made the proposed method suitable for the ultra-trace determination of PAHs.
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1. Introduction

Polycyclic aromatic hydrocarbons (PAHs), also known as polynuclear aromatic hydrocarbons or fused ring aromatic hydrocarbons, are a large group of persistent organic compounds (consisting of more than 100 compounds) with two or more fused aromatic rings and are listed as hazardous materials by the U.S. Environmental Protection Agency (EPA) and the World Health Organization (WHO). They are distributed worldwide in a variety of environmental matrices like air, water, and soil [1,2]. The incomplete combustion of fossil fuels and coal, petrochemical cracking processing, and asphalt and roofing activities are main sources of PAHs in the environment [3]. The degree of their acute toxicity is generally associated with the lower molecular weight PAHs and is correlated with aqueous solubility and octanol–water partition coefficients [4]. Exposure to PAHs has short- and long-term effects on human health. Some common symptoms of occupational exposure to PAHs are eye irritation, nausea, vomiting, diarrhea, confusion, skin irritation and inflammation [5]. PAHs may also cause long-term, chronic problems, such as cataracts, kidney and liver damage, and various types of cancer, including skin, lung, bladder, and gastrointestinal cancers [6].

In addition to environmental and occupational sources of exposure to PAHs, other important sources include smoked foods and smoking. Exposing foods to the smoke of burning wood are a traditional method of flavoring, cooking, and preserving them. It has been found, however, that smoking contaminates food items with various organic carcinogens, like PAHs [7]. The existence of PAHs in contaminated water and smoked foods has been proven using analytical techniques like gas and liquid chromatography [8–15].

Hookah, also known as water pipe, hubble–bubble, narghile (Turkish), or ghalyan (Persian), is a traditional instrument that has been used to smoke flavored tobacco in India, the Middle East, and central Asian countries for hundreds of years (Fig. 1). With the hookah, tobacco is burned on smoldering charcoal, and its smoke passes through water before being inhaled, exposing the smoker to dangerous amounts of PAHs [16].

Because of the well-known toxicity and hazards of PAHs to human health, levels of a variety of PAHs in the environment and

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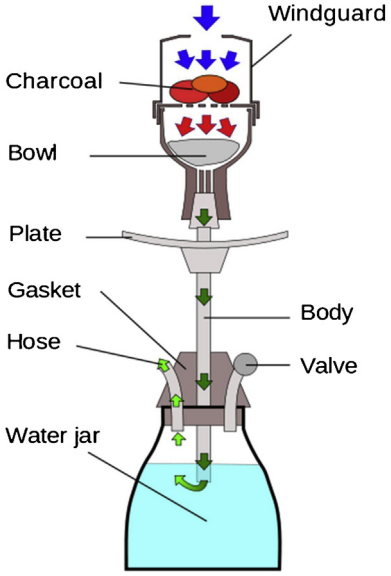

Fig. 1. Schematic structure of hubble–bubble.

in food are regulated by the EPA and the WHO [17]. There is a vital need for highly precise, accurate, and sensitive analytical methods for determining the levels of PAHs in various samples. Generally, PAHs have not been studied individually; therefore, chromatographic methods are superior to other methods for analyzing these compounds. High toxicity at low concentrations and different kinds of samples shows the necessity of efficient sample preparation techniques for clean-up and concentration as a part of the analytical process.

Solid-phase microextraction (SPME) was introduced by Arthur and Pawliszyn as a solvent-free sample preparation technique which integrates clean-up and preconcentration steps in chemical analysis [18]. After the introduction of nano-structured materials and the development of their application in various scientific and technological fields, the use of nano-sized sorbents as solid-phase microextraction fibers was presented [19–22]. Carbon nanotubes (CNTs) as one of the carbon allotropies were introduced by Iijima in 1991[23]. They have several formats such as single-walled (SWCNTs), multi-walled (MWCNTs), and functionalized CNTs. They possess superior mechanical, thermal, and electrical properties. Furthermore, their high porosity, hollow structure, large specific surface area, and light mass density are the prominent characteristics of adsorbent materials for extraction. The application of carbon nanotubes (CNTs) as the extracting phase of SPME fibers was reported by several research teams [24–43]. Alternately, hollow fiber membranes with a unique porous structure were used in different modes of liquid phase microextraction [44–46]. The application of hollow fiber membranes as SPME fiber was reported for the first time by us [47,48].

The present work deals with the application of a hollow fiber membrane as a porous bed for loading MWCNTs using a metallic wire as a mechanically durable support, and its application in the solid-phase microextraction of selected numbers of PAHs (five representative compounds) in samples of river water, hookah water (after smoking), and smoked rice.

2. Experimental

2.1. Chemicals and reagents

Naphthalene, phenanthrene, anthracene, fluoranthene, pyrene, tetrahydrofuran (THF), sodium chloride, and methanol were all obtained from Merck (Darmstadt, Germany). High molecular weight polyvinyl chloride (PVC) was purchased from FLUKA (Buchs, Switzerland). MWCNTs with an average external diameter of 8–10 nm were synthesized from acetylene on a Fe:Co catalyst at 720 °C using the chemical vapor deposition method [49]. The 50/280 Accurel polypropylene hollow fiber membranes (280 μm I.D.) from Membrana GmbH (Wuppertal, Germany) were used. Nitrogen and hydrogen (99.999% purity) were obtained from the Sabalan Oxygen Co. (Tehran, Iran). Stainless steel wire was purchased from Azar Electrode (Urmia, Iran).

Standard stock solutions of PAHs (100 mg L^{-1}) were prepared by dissolving appropriate amounts of them in methanol and storing them in darkness at 4 °C. Calibration series were prepared by diluting the stock solution in water daily; they were immediately used to prevent the sorption of analytes on the glass walls of the containers.

2.2. Apparatus

An Agilent 6890N, GC apparatus (Agilent Technologies, Wilmington, DE, USA) equipped with an FID detector and split/splitless injector was utilized for sample analysis. Separations were performed using an RTX-5 (5% diphenyl–95% dimethyl polysiloxane) capillary column (60 m × 0.25 mm I.D., film thickness 0.1 μm) (Restek, Bellefonte, PA, USA). ChemStation software (Agilent Technologies, Wilmington, DE, USA) was used to acquire and process data. A laboratory-made SPME device was used in all experiments. Coating surface morphology was studied using a JEOL scanning electron microscope, model JXA-840 (Tokyo, Japan), at Razi Metallurgy Research Center (Karaj, Iran). A Zag Shimi heater-stirrer (Tehran, Iran) was used to make temperature adjustments and for sample agitation.

2.3. Preparation of spiked rice samples

Samples of 100 g of milled rice were spiked with 1 mL of mixed working solution of the analytes (20 μg mL^{-1}). The spiked rice was allowed to dry at room temperature for 24 h and stored in darkness at −18 °C before the experiments.

2.4. Preparation of SPME fiber

The proposed fiber was prepared in three steps. First, a piece of stainless steel wire (1.5 cm length, 280 μm O.D.) was mounted on a SPME device. Next, it was entered into the narrow bore hollow fiber membranes (280 μm, I.D.), and any excess hollow fiber was cut. Lastly, 20 mg of MWCNTs was dispersed into a solution of 1 mg PVC powder in 5 mL THF and mixed well. The hollow fiber-coated steel wire was immersed in the suspension of MWCNTs in PVC solution for 50 min, and the MWCNTs were loaded into hollow fiber pores. The fiber preparation route is presented schematically in Fig. 2. Finally, the prepared fiber was conditioned at 150 °C for 30 min to remove any fiber contamination before use.

2.5. Solid-phase microextraction of PAHs from water and rice samples

The proposed fiber was exposed to the headspace of the water sample. During the extraction, the sample solution was stirred with a magnetic stir bar and the vial temperature was thermostated at 80 ± 1 °C using a water bath. Appropriate amounts of NaCl (3 mol L^{-1} concentration) were dissolved in the spiked aqueous solutions and real aqueous samples (river water and hubble–bubble water). After exposing the fiber to the headspace of the sample for 15 min, the fiber containing retained analytes was withdrawn from the vial and immediately inserted in the hot injection port of the GC, where the analytes were thermally desorbed at 150 °C for 5 min.

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