



A comparison study on a sulfonated graphene-polyaniline nanocomposite coated fiber for analysis of nicotine in solid samples through the traditional and vacuum-assisted HS-SPME

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ABSTRACT

A simple, rapid, and reliable headspace solid-phase microextraction (HS-SPME) procedure, reinforced by applying vacuum in the extraction vial, was developed. It was applied for the extraction of nicotine in solid samples prior to determination by gas chromatography-flame ionization detection (GC-FID). First, the surface of a narrow stainless steel wire was made porous and adhesive by platinization to obtain a durable, higher surface area, and resistant fiber. Then, a thin film of sulfonated graphene/polyaniline (Sulf-G/PANI) nanocomposite was synthesized and simultaneously coated on the platinized fiber using the electrophoretic deposition (EPD) method. It was demonstrated that the extraction efficiency remarkably increased by applying the reduced-pressure condition in the extraction vial. To evaluate the conventional HS-SPME and vacuum-assisted HS-SPME (VA-HS-SPME) platforms, all experimental parameters affecting the extraction efficiency including desorption time and temperature, extraction time and temperature and moisture content of sample matrix were optimized. The highest extraction efficiency was obtained at 60 °C, 10 min (extraction temperature and time) and 280 °C, 2 min (desorption condition), for VA-HS-SPME strategy, while for conventional HS-SPME the extraction and desorption conditions found to be 100 °C, 30 min and 280 °C, 2 min, respectively. The Sulf-G/PANI coated fiber showed high thermal stability, good chemical/mechanical resistance, and long lifetime. For analysis of nicotine in solid samples using VA-HS-SPME-GC-FID, linear dynamic range (LDR) was 0.01–30 $\mu\text{g g}^{-1}$ ($R^2 = 0.996$), the relative standard deviation (RSD%, $n = 6$), for analyses of 1 $\mu\text{g g}^{-1}$ nicotine was calculated 3.4% and limit of detection (LOD) found to be 0.002 $\mu\text{g g}^{-1}$. The VA-HS-SPME-GC-FID strategy was successfully carried out for quantitation of nicotine in hair and tobacco real samples.

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1. Introduction

Nicotine is a potent cholinomimetic stimulant and highly addictive and toxic alkaloid. It can be absorbed immediately in humans through the skin, mucosal lining of the mouth and nose or by inhalation in the lungs, which results in an increase in blood pressure and heart rate. Additionally, it is suspected to contribute to some known diseases like cancer. Thus, it has become increasingly important to develop new analytical methods for sensitive and selective determination trace amounts of nicotine in biological samples [1].

During the two recent decades, various solvent-free extraction platforms like solid-phase microextraction (SPME), as a proper alternative to the classical extraction methods, have been proposed.

SPME is as a fast, effective, and easily automated sample preparation strategy, but its commercial fibers are fragile, low-capacity and expensive [2]. Therefore, different new materials, coated on fused silica or metallic wires, were used for preparation of hand-made SPME fibers [3]. In this way, allotropic carbon nanoparticles were also used, due to their excellent thermal and chemical stability, very high specific surface area, and π - π electrostatic stacking interactions [4]. Graphene has a two-dimensional monolayer hexagonal lattice structure and possesses high specific surface area, chemical stability, ultra-high mechanical strength, and low preparation expense [5]. However, pure graphene irreversibly agglomerate and easily tend to curl. Thus, embedding appropriate functional groups to the graphene surface is necessary. Sulfonated graphene (Sulf-G) is one of the most interesting nanocomposites of graphene, because *p*-phenyl-SO₃H groups are located on the layers. It not only prevents the aggregation of the layers, but also improve the selectivity of graphene to extract distinctive analytes [6].

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Polyaniline (PANI) has established its applicability as a promising sorbent for analytical separation purposes, due to its unique properties [7]. Nonetheless, its traditional bulk synthesis produces irregular agglomerates with low specific surface area and poor processability. To improve these characteristics, its other nanostructures, with larger surface area, have been prepared through new synthetic strategies [8]. The stability and capability of these compounds is needed to be further amended, due to swelling and shrinkage of the conjugated backbone of PANI during its applications. The most effective approach for this purpose is preparation of PANI composites with carbon materials such as graphene. Graphene has low manufacturing cost and is a suitable support for PANI to introduce new sorbents, by combining the features of both compounds [9].

A serious challenge in the analysis of volatile organic compounds (VOCs) using headspace SPME sampling, is complete release of target analytes from the sample matrix and their effective trapping by the sorbent [10]. Different approaches have been reported to address this issue. One of the most efficient approaches to reduce the equilibrium time and reinforce the extraction efficiency is vacuum-assisted HS-SPME (VA-HS-SPME) [11]. The results of the VA-HS-SPME studies showed that reduced-pressure speeds up the release of analytes from the sample matrix and improve their partitioning into the headspace [12]. It was also revealed that the vacuum condition reduces the boundary layer around the fiber. This phenomenon reinforces analytes to trap onto the SPME fiber. It was also demonstrated that under-vacuum sampling significantly improves the extraction kinetics, especially for analytes with low Henry's constants. However, all reported VA-HS-SPME setups are closely similar [13]. In these systems the sample is directly exposed to headspace, during the evacuation process. This fact may impair the extraction process, due to sucking off liquid sample or solid particles inside the vacuum system. Additionally, solid samples are necessary to be mixed with water and handled as slurry mixtures. Therefore, presence large amounts of water can impair the extraction by increasing the number of competing molecules. Moreover, the vacuum vial must be removed and washed after each extraction. Consequently, the number of steps and time of the extraction process will be increased [14].

Based on the aforementioned descriptions, it seems to be important to design new improved VA-HS-SPME setups, in which these defects are resolved. In this way, a simple, low-cost, and easy-to-use VA-HS-SPME setup was designed and fabricated, which prevents the sample to be exposed to vacuum, during the evacuation process. It also provides the possibility to analyze both solid and liquid samples, without addition of water to create slurry. To evaluate the new device, a stainless steel wire was platinized and coated by Sulf-G/PANI nanocomposite using electrophoretic deposition (EPD) method. The prepared fiber was used to extract nicotine from tobacco and hair samples, followed by gas chromatography-flame ionization detector (GC-FID).

2. Experimental

2.1. Chemicals and materials

Aniline (99.5%), sodium dodecylsulfate (SDS) and analytical grade potassium hexachloroplatinate (IV) were purchased from Merck (Merck, Darmstadt, Germany). Analytical grade graphite, sulfuric acid, hydrochloric acid, nitric acid, sodium borohydride, hydrazine, sodium nitrite, potassium permanganate and hydrogen peroxide were provided by Merck. Nicotine (>99.0% analytical standard), were purchased from Sigma Aldrich (Sigma-Aldrich Chemie, Steinheim, Germany). Analytical reagent-grade sulfanilic acid, methanol and dichloromethane were purchased from Merck and

used without further purification. A stock solution (100 mg mL⁻¹) was prepared by dissolving an appropriate amount of nicotine standard in methanol. The working standard solutions were prepared weekly by diluting the stock solution with methanol. The standard and working solutions were stored at refrigerator. The tobacco sample, for preparation of the model matrix, was purchased from a local store in Khoramabad (Valiasr St., Khoramabad, Iran). The cigarette samples with commercial brand names of Kent (R.J. Reynolds Tobacco Company, Charlotte, NC, USA), Winston (Winston-Salem, R.J. Reynolds Tobacco Company, NC, USA) and Bahman (Iranian Tobacco Company, Tehran, Iran) were purchased from local stores in Khoramabad (Valiasr St., Khoramabad, Iran). The hair samples (a non-smoker man and two smoker men) were provided by Paliz Barber (Enghelab St., Khoramabad, Iran).

2.2. Instruments

Chromatographic analyses were performed using a Shimadzu GC-2010 Plus AF gas chromatograph (Shimadzu, Kyoto, Japan), equipped with a split/splitless injector (SPL-2010 Plus), a BPX-5 fused silica capillary column (30 m × 0.25 mm I.D., 0.25 μm), a FID-2010 Plus detector, and a GC solution software (version 2.4.1). A VEGA\TESCAN CM120-MIRA3 (TESCAN, Brno, Czech Republic) field-emission scanning electron microscope (FE-SEM) was employed to investigate the surface morphology of the Sulf-G/PANI nanocomposite. Fourier transform infrared (FT-IR) spectra were recorded using a Shimadzu FT-IR 8400 spectrometer and applied to characterize the functional groups on the sorbent. A PFO100 5RS Series ultrasonicator (18 kHz, 450 W, Sonica, Milano, Italy) was carried out for sonication. The stainless steel wire (0.01 mm O.D.) purchased from Vita Needle (Needham, MA, USA) and used as the fiber core. Heating the sample was performed using a MR 3001-K magnetic heater-stirrer (Heidolph, Kelheim, Germany). A 1-μL microsyringe (Hamilton, NV, USA) was used for direct injection GC-FID experiments. The VA-HS-SPME experiments were conducted using a 250 mL vacuum flask (ISOLAB, Wertheim, Germany) and 10-mL standard SPME vials from Supelco (Bellefonte, PA, USA). Commercial fibers of polydimethylsiloxane, 100 μm (PDMS), polyacrylate, 85 μm (PA), polydimethylsiloxane-divinylbenzene, 65 μm (PDMS/DVB), and stableflex divinylbenzene/carboxen/polydimethylsiloxane, 50/30 μm (DVB/CAR/PDMS) were provided by Supelco. A low-cost and a practically user-friendly hand-made SPME fiber holder was fabricated and used to utilize the fibers. For accurate and precise weight measurements, a Shimadzu AX-120 digital balance (Shimadzu, Kyoto, Japan) was used.

2.3. Gas chromatographic determination

To achieve the best separation condition, different exploratory temperature programs were examined. The best GC-FID temperature program was started at 100 °C (held 1 min) and then raised to 280 °C at a rate 20 °C min⁻¹ and held constant for 2 min. Accordingly, the total run time was 12 min. Both injector and FID detector were set at 280 °C. All GC-FID experiments were conducted in split mode (split ratio 1/10). High-purity nitrogen (>99.999%) was used as the carrier gas at a flow rate of 1 mL min⁻¹. The flow rates of nitrogen (makeup gas), hydrogen and air (FID gases) were 20, 30 and 300 mL min⁻¹, respectively. To obtain the retention time, the instrumental linear dynamic range and accurate quantification of nicotine, several standard solutions were directly injected into the GC-FID system and a calibration curve was constructed.

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