

***In-Situ* Intercalating of Silica Nanospheres into Polypyrrole During Its Electropolymerization to Prepare a Sorbent for Headspace Microextraction of Aldehydes in Edible Oils**

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Received: 09-29-2021

Abstract

The quality of edible oils is significantly affected by autooxidation of lipids, which alters their flavor and nutritional quality through production of toxic materials like aldehydes (an important class of oil deterioration markers). Herein, an amino-silica nanospheres/polypyrrole (ASNS/PPy) nanocomposite sorbent was synthesized and used as the fiber coating for headspace solid-phase microextraction (HS-SPME) of aldehydes in edible oils, followed by gas chromatography (GC) separation and determination. Amino-silica nanoparticles were prepared by an amended Stöber method and composited with polypyrrole during its electropolymerization on the surface of a platinized stainless-steel fiber. The synergy between *in-situ* electropolymerization and rough surface of the platinized metal substrate created a durable fiber coating with unique uniformity, cohesiveness, and adsorption properties. The synthesized nanocomposite was characterized using Fourier transform infrared spectroscopy and scanning electron microscopy techniques. The performance of the prepared fiber was optimized by investigating the affecting variables including extraction temperature and time, stirring rate, and desorption conditions. The obtained limits of detection for hexanal and heptanal in sunflower oil were 0.005–0.009 $\mu\text{g mL}^{-1}$. The prepared fiber exhibited excellent repeatability and reproducibility with the intra-fiber and inter-fiber relative standard deviations in the ranges of 3.9–8.8% and 7.3–15.1%, respectively. The proposed HS-SPME-GC strategy was successfully applied for the analysis of aldehydes in commercial edible oil samples.

Keywords: Amino-silica nanospheres; polypyrrole nanocomposite; headspace solid-phase microextraction; edible oil; aldehydes.

1. Introduction

Researchers have recently paid more attention to edible oils because of their importance in human health and nutrition. The quality of edible oils depends on their origin, processing, and oxidation state. The main edible oil components are saturated and unsaturated fatty acids.¹ Oxidation is the main reason for the instability of edible oils and causes a change in chemical and nutritional properties of oils. Oxygen in the air causes unsaturated fatty acids of edible oil to undergo non-enzymatic self-oxidation, leading to the production of peroxides.^{2,3} Aldehydes are an important class of lipid oxidation products, well-known for their health risks. They are a major cause of unpleasant odor of rancid oil. Therefore, quantification of aldehydes (as chemical markers) in edible oils is of great

importance for quality control purpose. There is a wide range of saturated and unsaturated aldehydes and ketones in oxidized edible oils, however, short chain aldehydes like hexanal and heptanal are more abundant. Therefore, these compounds are the most used biomarkers for the quality control of edible oils, due to their higher concentrations in oxidized edible oils. Despite huge advances in chemical analysis, sample pretreatment of edible oils for oxidation stability studies is still mostly performed using classical extraction methods like solvent extraction and Soxhlet which are tedious, time-consuming, and expensive. Moreover, these methods usually use toxic organic solvents and reactive chemical compounds which can expose operators and/or samples to hazardous materials.⁴ Accordingly, development and application of novel and green methods for chemical analysis of edible oils is now a necessity.

Solid-phase microextraction (SPME) is a green solvent-free sample pretreatment technique, demonstrated to be a promising method for the sampling of organic compounds in food, biological, and environmental samples.⁵ SPME can be easily coupled with different chromatographic techniques, automated, and portabilized. The most widely used mode of SPME is headspace (HS), which extracts analytes from the upper atmosphere of the sample, without direct contact with the sample matrix. HS-SPME is the best choice for the sampling of volatile and semi-volatile organic compounds. That is why HS-SPME has been widely used for the sampling of odors, flavors, and fragrances in food samples.^{1,6} Extensive efforts have been devoted to expanding applicability and improve performance of SPME, particularly by preparation of new sorbents using nanomaterials. Compared to the commercial sorbents, nanomaterial-based sorbents benefit from higher porosity, larger surface-to-volume ratio, better thermal and chemical stability, more selectivity, and higher extraction efficiency.⁵ Among these, silica nanoparticles have received more attention due to their abundance, cheapness, various available surface modification methods, high porosity and adsorption power, and non-toxicity.^{7–9} To improve the extraction efficiency and selectivity, silica nanoparticles have been composited with different materials like TiO₂,¹⁰ graphene oxide (GO),¹¹ Fe₃O₄,¹² and conductive polymers.¹³ Among conductive polymers, polypyrrole (PPy) and its derivatives have attracted much consideration in SPME techniques due to their significant advantages including non-toxicity, low-cost, thermal stability, and particularly simple synthesis routes.¹⁴ As a unique and promising feature in the preparation of SPME sorbents, thin layers of PPy with controllable properties can be coated on conductive surfaces like stainless-steel through *in-situ* electropolymerization during electrophoretic deposition (EPD) from different aqueous or organic solutions.¹⁵ For this reason, different composites of PPy like graphene/polypyrrole,^{16,17} magnetite nanoparticles/polypyrrole,¹⁸ multiwalled carbon nanotubes/polypyrrole,¹⁹ functionalized graphene and ionic liquid co-doped polypyrrole,²⁰ and iron oxide/silica/polypyrrole²¹ have been successfully employed for the preparation of SPME fibers. The efficiency of PPy composites (e.g., polypyrrole/graphene oxide) for the extraction of aldehydes in edible oil has been previously demonstrated through a reduced-pressure fiber-in-needle technique.²²

In this research, amino-silica nanospheres (ASNS) were synthesized and composited with PPy during its *in-situ* electropolymerization on the surface of a stainless-steel fiber. The ASNS/PPy coated fiber was applied for the HS-SPME sampling of aldehydes (hexanal and heptanal) in edible oils. The analytes were quantified using gas chromatography-flame ionization detection (GC-FID), without derivatization. To the best of our knowledge ASNS/PPy has not been previously reported as a SPME fiber coating.

2. Materials and Methods

2.1. Chemicals

Analytical reagent grade aldehydes were obtained from Merck (Darmstadt, Germany). Stock standard solutions (1000 mg L⁻¹) of hexanal (Hex) and heptanal (Hep) were prepared in methanol and stored at 4 °C. Working standard solutions were prepared by dilution of the stock solution in double distilled water, on a daily basis. Pyrrole (> 99%) was provided by Fluka (Buchs, Switzerland). Pyrrole was redistilled and stored in a dark bottle under nitrogen atmosphere in a refrigerator before each use. Tetraethyl orthosilicate (TEOS, 98%), succinic anhydride, *N,N*-dimethylformamide (DMF) and, *N,N*-dicyclohexylcarbodiimide (DCC) were purchased from Merck. Analytical grade potassium hexachloroplatinate(IV) (K₂PtCl₆), ethanol, methanol, NaNO₃, NaOH, LiClO₄, concentrated hydrochloric acid (HCl), and aqueous ammonia (NH₃,aq) solutions were purchased from Merck.

2.2. Gas Chromatographic Analysis

A Shimadzu GC-2010 Plus AF gas chromatograph (Shimadzu, Kyoto, Japan), equipped with a split/splitless injector (SPL-2010 Plus) and a flame ionization detector (FID-2010 Plus), was used for the gas chromatographic separations and determinations. It used GC solution software (version 2.4) and a BP-5 fused-silica capillary column (30 m × 0.25 mm × 0.25 µm). Ultra-high purity nitrogen was used as the carrier gas with a constant flow rate of 1 mL min⁻¹. All SPME injections were performed in splitless mode. The initial column temperature was set at 60 °C and then increased to 100 °C at a rate of 4 °C min⁻¹. Finally, it was ramped to 280 °C at a rate of 70 °C min⁻¹. The GC detector and injector were maintained at 300 and 280 °C, respectively. Hydrogen and zero-air were used as the FID gases at flow rates of 30 and 300 mL min⁻¹. Nitrogen was used as the make-up gas with a flow rate of 30 mL min⁻¹. Fourier transform infrared (FT-IR) spectra were obtained in the transmittance mode, by a Shimadzu FT-IR 8400 spectrometer. Scanning electron microscopy (SEM) micrographs were recorded using a CM120 Vega field-emission scanning electron microscope (TESCAN, Brno, Czech Republic), to investigate the surface morphology of the nanocomposite.

2.3. Preparation of ASNS/PPy Coated SPME Fiber

Silica nanospheres was prepared through hydrolysis of TEOS according to an amended version of Stober's method.⁹ For synthesis of ASNS, 20 mg of silica nanospheres was dispersed in 20 mL of DMF in an ultrasonic bath. Then, 20 mL of succinic anhydride solution (0.1 M) was added dropwise, and the suspension stirred for 24 h. Finally, the precipitate was centrifuged, rinsed with DMF, and dried in a vacuum oven at 60 °C for 4 h.

To create a porous, high surface area and robust fiber substrate, the surface of stainless-steel fiber (4 cm length and 0.075 mm i.d.) was platinized based on an EPD method.²³ Then, the fiber was coated by ASNS/PPy nanocomposite *via* intercalating of ASNS in PPy during electropolymerization of pyrrole, using a two-electrode electrochemical system. To this end, 0.08 g ASNS was dispersed in 10 mL pyrrole solution (containing 0.1 M LiClO₄). The platinized fiber was connected to the anode and a normal stainless-steel wire was connected to the cathode of a DC power supply and immersed into the suspension. The distance between the electrodes was 2.0 cm and a 1.0 V DC voltage applied to the electrodes for 20 min. Then, the fiber was removed from the electrochemical cell and rinsed with water and ethanol and dried. Before the first use the fiber was preconditioned under nitrogen atmosphere for 30 min at 280 °C.

2. 4. HS-SPME Sampling of Aldehydes in Edible Oils

Using a blank sample matrix (model matrix) is essential to optimize an extraction method in a reliable way. This fact is more serious for the analysis of complex sample matrices like edible oils. In this research, a liquid oil sample was used as the model matrix, after being processed to remove its aldehyde content. For this purpose, 500 mL of sample oil was poured into a 1000-mL vacuum container and subjected to vacuum for 20 min at 25 °C along with continuous stirring. Under the vacuum, aldehyde contents were removed from the oil matrix, resulting in a model matrix without aldehydes. It was stored at 4 °C in a fridge under nitrogen atmosphere to prevent further oxidation and production of aldehydes. The absence of aldehydes in the model sample was checked by using GC analysis. To optimize the parameters affecting the extraction efficiency, 3 mL of model sample was transferred to a 10 mL SPME vial and spiked with known concentrations of the analytes. Then, the vial was capped, and the sample was homogenized by sonication for 2 min. After that, the extraction vial was placed into a water bath on a hotplate-magnetic stirrer at 50 ° and stirred at 750 rpm. The ASNS/PPy fiber was mounted on a handmade fiber holder and exposed to the headspace of the sample for 15 min. Finally, the fiber was retracted and immediately injected into the GC injection port for thermal desorption of the extracted analytes at 280 ° for 1 min.

3. Results and Discussion

Regardless of the physicochemical stability of the coating and fiber substrate, a serious problem in commercial and homemade SPME fibers is weak adhesion between the coating and the fiber substrate. Hence, the coating is easily stripped or exfoliated from the substrate after expo-

sure to high temperature, organic solvents, and/or mechanical tension. Two complementing strategies have been suggested to address this problem, the first chemical bonding of the coating to the substrate²⁴ and the second roughening the surface of the substrate.²⁵ Fortunately, to prepare industrial protective and anticorrosion coatings for stainless-steel extensive studies have been done and deep knowledge developed.²⁶ For example, Vasconcelos *et al.*²⁷ prepared a very highly thermally stable and substantially corrosion resistant coating for stainless-steel using silica thin-films through a sol-gel technique. Accordingly, in this research, the surface of the stainless-steel fiber was platinized to create a rough and increased surface area substrate. The effectiveness of this method has been well proven in our previous studies.^{9,23,28} This research tried to fabricate a very durable SPME fiber by electrodeposition of ASNS/PPy on the rough surface of platinized stainless-steel fiber.

3. 1. Characterization of ASNS/PPy Nanocomposite

To characterize the chemical bonds in the structure of SNS, ASNS, and ASNS/PPy, their FT-IR spectra were recorded (Fig. 1). The absorption peak at 1099 cm⁻¹ (indicating O-Si-O bond) and the peaks appearing at 802.33 and 470.60 cm⁻¹ (correspond to the symmetric stretching and rocking vibrations of Si-O-Si, respectively) show the polymeric structure of silica. The presence of N-H bond in the ASNS structure can be clearly inferred from the vibrational peak 1635 cm⁻¹. While ASNS/PPy nanocomposite characteristic peaks can be seen at 1099 cm⁻¹ (O-Si-O bond), 1554 cm⁻¹ (N-H and C-N bonds), 1635 cm⁻¹ (N-H bond), 1658 cm⁻¹ (C=O bond), 2927 cm⁻¹ (C-H bond), and 3417 cm⁻¹ (O-H bond).²⁹

SEM micrographs of ASNS and ASNS/PPy nanocomposite were recorded to evaluate their morphological surface features, as depicted in Fig. 2. The spherical configuration of the silica nanospheres and their particle size uniformity are obvious in Fig. 2a. Additionally, conical pillars of PPy can be clearly observed in Fig. 2b and 2c. These conical pillar structures can be considered as the grown (thickened) forms of PPy nanowires, as has been shown previously.³⁰ Mechanical strength of the coating was tested by scraping the fiber coating using a knife to simulate its possible damage by the edge of the protecting needle during the injection/withdraw cycles.³¹ It was found that the ASNS/PPy coating was difficult to be scraped using a knife. The thermal stability of the fiber was demonstrated by performing up to 90 extractions, each followed by a thermal desorption step in the GC injector at 280 °C. To evaluate the stability of the ASNS/PPy coated fiber against organic solvents, it was directly immersed in ethanol, acetone, acetonitrile, hexane, and methanol for 30 min. Then, the fiber was rinsed with water and conditioned in GC injector for 10 min at 250 °C to

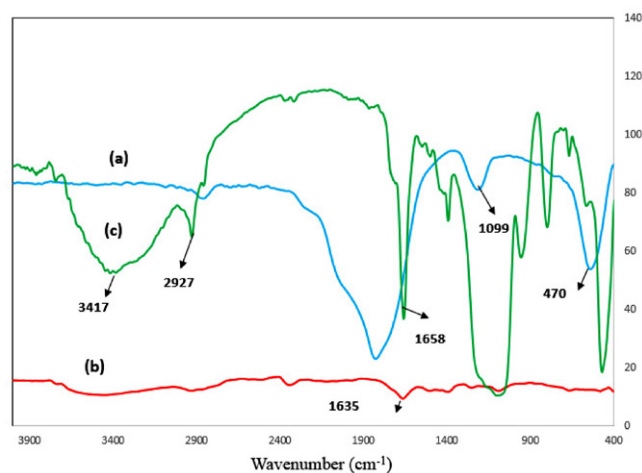


Fig. 1. FT-IR spectra of SNS (a), ASNS (b), and ASNS/PPy nanocomposite (c).

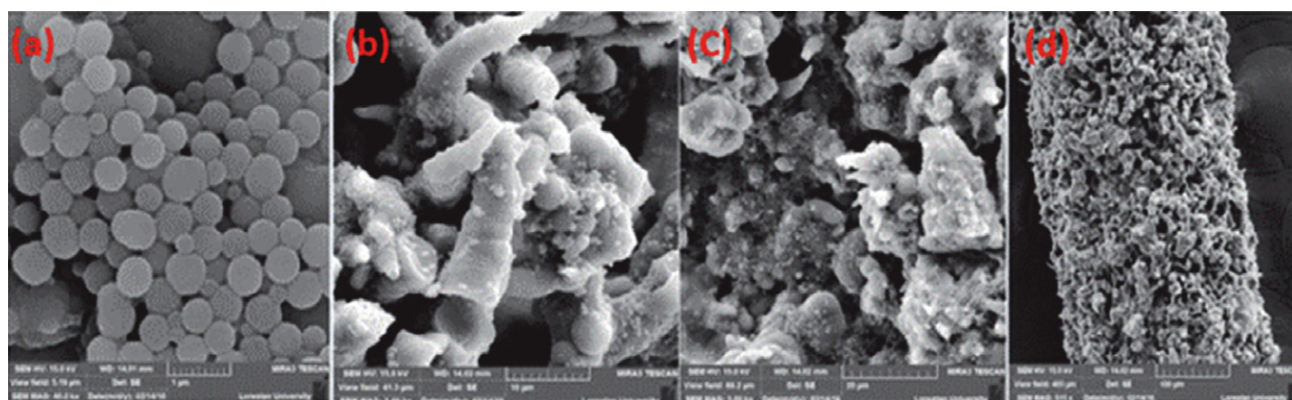


Fig. 2. SEM micrographs of (a) SNS, (b and c) ASNS/PPy nanocomposite at different magnifications, and (d) the ASNS/PPy coated stainless-steel fiber.

remove any possible solvent residues, before extracting the analytes. The results demonstrated that the fiber was resistant to organic solvents and the extraction efficiency didn't change significantly (< 5%).

3. 2. Optimization of the Extraction Conditions

To achieve the highest extraction efficiency, the important influential variables including desorption time, desorption temperature, sampling temperature, sampling time, and stirring rate were optimized. To ensure the complete transfer of the extracted analytes from the fiber to the chromatographic column and acquire accurate results, desorption conditions must be optimized first. Accordingly, desorption temperature and desorption time were investigated in the ranges of 200–300 °C and 1–4 min, respectively. The highest peak areas were observed at desorption temperatures more than 280 °C and at desorption time of 1 min, with the minimum carryover effect (< 5%). Therefore, 280 °C and 1 min were selected as the optimal desorption conditions for further investigations.

3. 2. 1. Effect of Sampling Temperature

Extraction temperature is one of the main influential parameters affecting the equilibration of analytes between the headspace and fiber coating, and consequently on the extraction efficiency, particularly for volatile analytes like short-chain aldehydes. Higher temperatures improve the release of analyte from the sample matrix and increase their concentrations in the headspace, leading to an increase in the extraction amounts. Simultaneously, temperature of the fiber coating also rises and reduces the analyte trapping, due to the exothermic effect of adsorption process. For this reason, HS-SPME sampling temperature needs to be optimized carefully.³² Therefore, sampling temperature was evaluated over the range of 40–90 °C. As can be seen from the results (Fig. 3a), the peak areas of the analytes increased by raising sample temperature. This fact can be explained by the formation of more aldehydes by raising the sample temperature (due to increased lipid oxidation), as substantiated in previous investigations.²² Therefore, it was better to select a temperature from the most stable part of the graph (Fig. 3a), with minimal fluctuations of response with temperature change. Therefore,

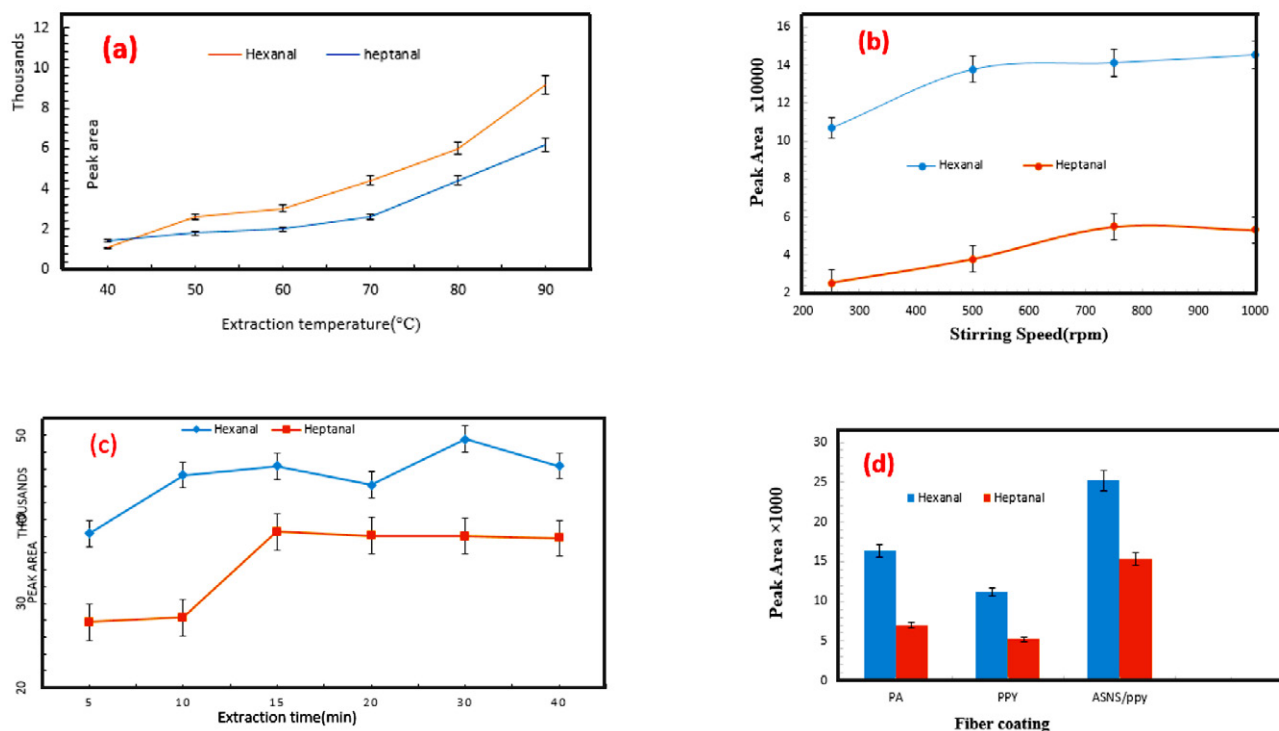


Fig. 3. Dependence of the extraction efficiency of the developed HS-SPME method on sample temperature (a), stirring speed (b), and extraction time (c). Comparison of the ASNS/PPy coated fiber with commercial polyacrylate fiber and a homemade PPy fiber (d), under the optimized conditions (extraction time, 20 min; stirring rate, 600 rpm; desorption temperature: 280 °C; and desorption time, 1 min; repeated three times).

50 °C was chosen as the optimal HS-SPME sampling temperature.

3. 2. 2. Effect of Sample Stirring Speed

Increasing the stirring speed in HS-SPME improves the mass transfer and increases concentration of analytes in the sample headspace. Furthermore, the time required to achieve thermodynamic equilibrium is reduced. To evaluate this effect, stirring speed was varied over the range of 200–1000 rpm and GC-FID peak areas of the analytes were recorded. The results depicted in Fig. 3b show that the peak areas for both analytes increase with raising the stirring speed up to 750 rpm and then remain nearly constant. So, 750 rpm was chosen as the optimal stirring speed for further studies.

3. 2. 3. Effect of Sampling Time

To achieve the highest extraction efficiency, the extraction time was examined in the range of 5–40 min. As

shown in Fig. 3c, peak areas of both analytes increased by prolonging the extraction time up to 15 min and then remained constant. Thus, 15 min was chosen as the extraction time for further experiments.

3. 3. Analytical Performance

To evaluate the quantitative analytical figures of merit of the developed method, its linear dynamic ranges (LDRs), limits of detection (LODs), and relative standard deviations (RSDs) for the extraction of Hex and Hep from edible oil samples were investigated, under the optimized conditions. The calibration graphs were linear over the range of 0.03–20 $\mu\text{g mL}^{-1}$ for hexane and 0.05–25 $\mu\text{g mL}^{-1}$ for heptanal, with linear regression coefficients greater than 0.998. The LODs correspond to the analyte amounts for which the signal-to-noise ratio was equal to 3, and were found to be 0.005 for hexanal and 0.009 $\mu\text{g mL}^{-1}$ for heptanal. The precision was assessed by obtaining the RSDs including repeatability (intra-fiber) and reproduc-

Table 1. Analytical figures of merit of the HS-SPME-GC procedure for the sampling and quantification of hexanal and heptanal in edible oil.

Analyte	Equation	R^2	LDR ($\mu\text{g mL}^{-1}$)	LOD ($\mu\text{g mL}^{-1}$)	RSD% (n = 6)	
					Intra-fiber	Inter-fiber
Hexanal	$y = 1139.4x + 5807.1$	0.9989	0.03–20	0.005	3.9	7.3
Heptanal	$y = 209.09x + 1586.2$	0.9982	0.05–25	0.009	8.8	15.1

ibility (inter-fiber), which varied between 3.9–8.8% and 7.3–15.1%, respectively (Table 1). A single fiber was used for more than 90 extractions (extraction/desorption cycles) without significant change (< 5%) in its extraction efficiency.

To evaluate reliability and applicability of the ASNS/PPy coated fiber, it was compared with a commercial PA coated fiber and a homemade PPy coated fiber, under the optimized conditions (Fig. 3d). The results demonstrated that the ASNS/PPy fiber was remarkably more efficient than the tested fibers for the extraction of the aldehydes in edible oils.

3. 4. Comparison of the Proposed Method with Similar Reported Procedures

To further indicate the supremacy of the proposed microextraction strategy, its significant features were compared with some of the similar reported microextraction methods.^{22,33–36} As can be seen in Table 2, the proposed procedure shows wider linear ranges and acceptable RSDs, compared to most of the listed procedures. Additionally, the LODs are lower than most of the reported methods, even compared to the method that used a MS detector.³⁶ However, its LODs and LDRs are not comparable with the

reduced-pressure fiber-in-needle method (RP-FIN) technique, which has used a low-pressure dynamic microextraction strategy.²² Anyhow, the proposed method uses a low-cost and durable fiber and instrumentation (GC-FID), without any derivatization methods.

3. 5. Determination of Hexanal and Heptanal in Edible Oils

To test the applicability of the developed method, it was applied for the determination of hexanal and heptanal in three commercial sunflower oil samples of different brands, produced at different dates. Samples 1, 2, and 3 had been produced 35, 36, and 4 months before the analysis time, respectively. The samples had been stored unopened at the same conditions. To ensure the reliability of the HS-SPME-GC-FID strategy, each sample was also spiked with $1 \mu\text{g mL}^{-1}$ of the analytes and subjected to the analysis, three times. As shown by the results in Table 3, acceptable recoveries and RSDs were obtained, indicating that the developed method can be successfully applied for the quantification of aldehydes and study of oxidative stability of edible oils. Fig. 4 shows a typical GC-FID chromatogram of a sunflower oil, analyzed by the developed HS-SPME-GC-FID method.

Table 2. Comparison of the developed HS-SPME-GC procedure with some similar microextraction procedures for the analysis of aldehydes in edible oils.

Method	Extraction phase	LDR ($\mu\text{g mL}^{-1}$)	LOD (ng mL^{-1})	Intra-fiber RSD (%)	Inter-fiber RSD (%)	Matrix	Ref.
USABE-GC-FID ^a	Triton X-114 in CHCl_3	0.05–20	20–150	2–4	–	Heated soybean, sunflower, and olive oils	35
HS-SPME-GC-FID	PDMS/CAR	0.05–10	5–10	8.9	–	Peanut, soybean, and olive oils	36
HS-SPME-GC-FID	DVB/CAR/PDMS	–	160–350	4.02–8.53	–	Sunflower and rapeseed oils	37
SPME-GC-MS	PDMS/PEG	0.02–1	6–15	8.9–11.2	9.3–16.2	Sunflower oil	38
RP-FIN-GC-FID ^b	PPY/GO	0.0007–50	1–8	5.9–9.0	–	Corn, sunflower, soybean, olive, canola, and sesame oils	24
HS-SPME-GC-FID	ASNS/PPy	0.03–25	5–9	3.9–8.8	7.3–15.1	Sunflower oil	This work

^a Ultrasound-assisted back extraction reverse micelles method coupled with GC-FID. ^b Reduced-pressure fiber-in-needle coupled with GC-FID.

Table 3. HS-SPME-GC-FID sampling and determination of hexanal and heptanal in commercial edible oil samples using the ASNS/PPy coated fiber.

Edible oil	Added ($\mu\text{g mL}^{-1}$)	Found ($\mu\text{g mL}^{-1}$)		Recovery (%)	
		Hexanal	Heptanal	Hexanal	Heptanal
Sample# 1	0	0.67	1.79	–	–
	1	1.58	2.73	90	93
Sample# 2	0	9.34	8.99	–	–
	1	10.28	9.87	94.5	88.5
Sample# 3	0	0.56	0.09	–	–
	1	1.43	0.93	86.8	84.2

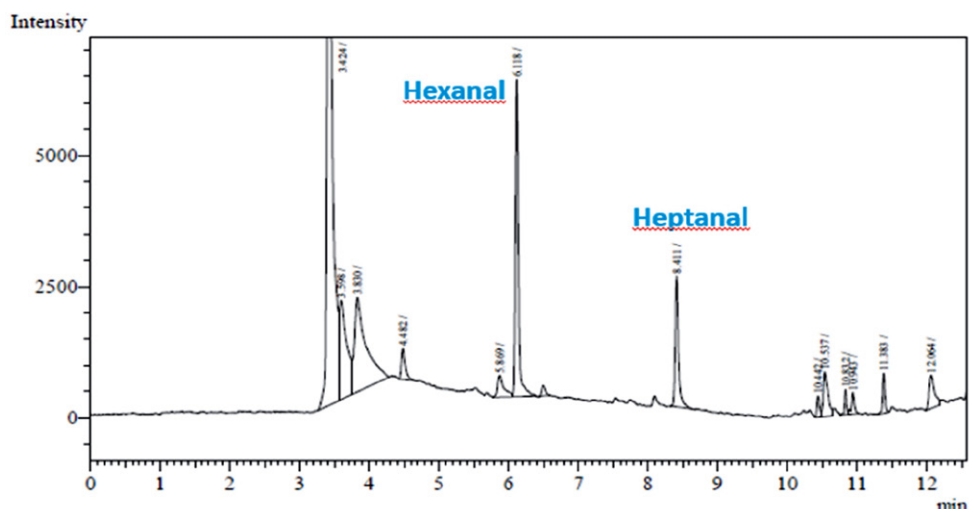


Fig. 4. A GC-FID chromatogram of a sunflower edible oil sample after HS-SPME sampling using an ASNS/PPy coated fiber.

4. Conclusions

Amino-functionalized silica nanospheres/polypyrrole (ASNS/PPy) composite was synthesized, characterized, and employed to prepare a SPME fiber. First ASNS was synthesized *via* a green sol-gel procedure. They were then dispersed into a pyrrole solution, in a two-electrode electrochemical cell, and coated on the surface of a platinized stainless-steel fiber, during electropolymerization of polypyrrole. By combining the features of a platinized metallic surface and an *in-situ* electropolymerization, a highly porous and durable SPME fiber was obtained. The sorption efficiency and durability of the fiber was confirmed by successful extraction of aldehydes from edible oil samples. The results demonstrated that the developed fiber and methodology can be effectively applied for the sampling and trapping of volatile and semi-volatile analytes in food, biological, and environmental samples.

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Povzetek

Na kakovost jedilnih olj znatno vpliva avtooksidacija lipidov, ki spremeni njihov okus in prehransko kakovost zaradi nastanka strupenih snovi, kot so aldehydi (pomemben razred označevalcev razkroja olja). V našem primeru smo sintetizirali nanokompozitni sorbent: nanokroglice iz amino-silicijevega dioksida/polipirola (ASNS/PPy), ki smo ga uporabili kot prevleko za vlakno za mikroekstrakcijo na trdno fazo iz nadprostora (HS-SPME) aldehydov iz jedilnih olj, sledila pa je ločba in določitev s plinsko kromatografijo (GC). Nanodelci iz amino-silicijevega dioksida so bili pripravljani po prirejeni Stöberjevi metodi in skupaj s polipirolom z elektropolimerizacijo deponirani na površino platiniziranega vlakna iz nerjavečega jekla. Sinergija med *in-situ* elektropolimerizacijo ter grobo površino platiniziranega kovinskega substrata je ustvarila odporno prevleko vlakna z edinstveno enakomernim nanosom, kohezivnostjo in adsorpcijskimi lastnostmi. Sintetizirani nanokompozit smo okarakterizirali z infrardečo spektroskopijo s Fourierjevo transformacijo ter z vrstično elektronsko mikroskopijo. Učinkovitost pripravljenega vlakna smo optimizirali s spreminjanjem pogojev, kot so temperatura in čas ekstrakcije, hitrost mešanja in pogoji desorpcije. Dosežene meje zaznave za heksanal in heptanal v sončničnem olju so bile 0,005–0,009 $\mu\text{g mL}^{-1}$. Pripravljeno vlakno je imelo odlično ponovljivost in obnovljivost z relativnimi standardnimi odmiki 3,9–8,8 % za eno vlakno ter 7,3–15,1 % med vlakni. Predlagano HS-SPME-GC metodo smo uspešno uporabili za analizo aldehydov v komercialnih vzorcih jedilnih olj.



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