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Determination of Benzo[a]pyrene in Edible Oil by Nickel Oxide Deposited Silica Based Solid-Phase Extraction Combined with High Performance Liquid Chromatography-Diode Array Detector

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Determination of Benzo[a]pyrene in Edible Oil by Nickel Oxide Deposited Silica Based Solid-Phase Extraction Combined with High Performance Liquid Chromatography-Diode Array Detector

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Abstract

A simple, rapid, and cost-effective method for the determination of BaP in edible oil was developed and validated. Nickel oxide deposited silica (SiO_2@NiO) prepared by depositing nickel oxide onto silica using liquid phase deposition method was employed as solid-phase extraction (SPE) adsorbent for the extraction of benzo[a]pyrene (BaP) in edible oil followed by high performance liquid chromatography-diode array detector (HPLC-DAD) analysis. The edible oil was diluted with n-hexane and then directly loaded to SiO2@NiO for SPE. The n-hexane was also used to clean the fat-soluble interference in the edible oil, while BaP was selectively captured due to the electron donor-acceptor interaction with SiO_2@NiO. The extraction conditions such as amount of sorbent, volume of washing solvent, type and volume of desorption solvent were optimized. The method demonstrated good linearity over the range of 6-1875 ng/g with the limit of detection of 1.3 ng/g, the spiked recoveries in the range of 97.4-105.1 %, and the relative standard deviation (RSD) less than 3.0 %. The method was applied for the analysis of BaP in 12 actual oil samples and the results showed that unrefined oil and high-temperature frying oil were at risk of BaP exceeding the acceptable level.

Keywords: nickel oxide deposited silica; benzo[a]pyrene; high performance liquid chromatography-diode array detector; edible oil

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1. Introduction

Benzo [a] pyrene (BaP) is a polycyclic aromatic hydrocarbon with high molecular weight composed of pyrene and phenylene and was recognized as a marker for carcinogenic PAHs in food [1,2]. It often entered the human body mainly by eating the foods contaminated with BaP [3–6]. Edible oils are necessary in People's Daily diet and contain rich triglycerides, fatty acids, fat soluble vitamins and other nutrients [7,8]. They were highly vulnerable to contamination when produced using BaP -contaminated crops or by incorrect processing methods [9–11]. In addition, the high temperature during frying can also lead to the formation of BaP [12–14]. Therefore, to reduce the risk to human health, permissible level of BaP in edible oil has been set by legislative requirements. For example, the maximum residual amount of BaP in cooking oil was set at 10 ng/g by GB 2762-2017 [15]. So it was necessary to monitor BaP in edible oil quantitatively.

Besides, it was a challenge to develop a simple, low-cost, novel and selective quantification method of BaP in edible oils. Due to the inexpensive and stable detected performance, high-performance liquid chromatography coupled with diode array or ultraviolet detector (HPLC-DAD/UV) was accessibly used [15,16]. However, it was difficult to detect BaP because of its low

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content of BaP in edible oils with complex composition [16]. Therefore, suitable sample pretreatment methods to purify and enrich BaP from edible oils were necessary before instrumental analysis [17,18]. Solid phase extraction (SPE) was a widely used sample pretreatment technology, characterized by simple operation, good reproducibility and strong enrichment ability. It achieved the capture and enrichment of the target analyte via the selective interaction between the adsorbent and the target analytes, so the adsorbent was the key of SPE [19–23]. However, given that BaP and edible oil matrix both possessed high lipophilicity, it is difficult to selectively extract BaP from edible oil by conventional solid phase extraction column (such as C_{18}) based on hydrophobic interaction, so it is necessary to develop new adsorbents with high extraction selectivity for BaP in edible oil [24]. In our previous work [25], humic acid modified silica gel adsorbent was prepared and used for selective enrichment of BaP in edible oil through π-π interaction. Guo et al. [26] prepared a tetraoxacyclix [2] arene [2] triazine solid phase extraction adsorbent, and established an analytical method for the detection of BaP in edible oil based on the complex mechanism. However, the preparation processes of the above adsorbents were too complex and required multi-step synthesis reaction.

Liquid phase deposition (LPD) was a green and simple method for preparing oxide coatings, which could uniformly deposit different nano-oxide coatings on various substrates [27]. Various metal oxide coatings had been prepared by LPD and used in separation science [27–30]. Among metal oxides, NiO related adsorbents exhibited high selectivity for imidazoles based on the coordination interaction between Ni(Ⅱ) in the material and imidazole groups [30–33]. Nevertheless, It had not been used as an adsorbent to selectively extract BaP from a hydrophobic matrix such as edible oil. The possible charge - transfer interaction between Ni((Ⅱ) with vacancies and BaP with rich π electrons makes NiO a potential adsorbent for the selective extraction of BaP.

In this work, nickel oxide nanoparticles (NiO) was simply deposited on silica by liquid phase deposition (LPD) method and then used for SPE of BaP in edible oil. Finally, a simple, selective and reliable method to determine BaP in edible oils was established.

2. Materials and Methods

2.1. Chemicals and Reagents

Dichloromethane, n-hexane, acetone, acetonitrile, methanol and ethanol were purchased from Wuhan Freton Co., Ltd. (Wuhan, China). Nickel fluoride tetrahydrate ($NiF_2·4H_2O$), boric acid (H_3BO_3) and hydrochloric acid (HCl) were from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Benzo(a)pyrene (BaP) was obtained from YuanYe Biotechnology (Shanghai, China). Purified water was obtained with Milli-Q apparatus (Bedford, MA, Millipore). The silica (200-300 mesh) used for preparing the SPE adsorbent was obtained from Qingdao Marine Chemical Plant (Qingdao, China).

2.2. Equipment and Chromatographic Analysis Conditions

Field emission scanning electron microscope (FE-SEM, Germany, Zeiss Merlin Compact) with EDS mapping (X-MaxN). The specific surface areas and pore sizes were obtained by the results of nitrogen adsorption analysis with a TriStar Ⅱ 3020 specific surface area analyzer from Micromeritics (Norcross, GA, USA). The HPLC system used was LC-20A from Shimadzu (Japan) equipped with a quaternary pump and a diode array detector (SPD-M20A). Separation of BaP in edible oils were conducted using a Inertsil ODS-4 column (150 mm×4.6 mm; GL Sciences, Japan) at room temperature, with an injection volume of 20 μL. The mobile phase consisted of acetonitrile and water (80/20 v/v) at flow rate of 1.0 mL/min. The detection wavelength was set 295 nm.

2.3. Preparation SiO_2@NiO

According to the previous work of our group [29], the SiO_2@NiO material was prepared by the LPD method. Simply, a certain amount of silica ((200 - 300 mesh) was put into a 1:1 (v/v) hydrochloric acid aqueous solution and stired for 24 h to remove metal ions from the surface of the material. The treated silica was washed in deionized water until neutral, then dried.

After fully mixing 15 mL of saturated NiF_2 aqueous solution and 25 mL of 0.6 mol/L H_3BO_3 aqueous solution, 2.0 g of activated silica was added, and the mixture was shaken at 40 ℃ for 72 h. The material was filtered, washed with deionized water, and dried at 60 ℃.

Finally, it was calcined at 200 ℃ for 1 h in an SX2-2.5-10 Muffle furnace from Jianli Electric Furnace Manufacturing Co., Ltd., (Yingshan, China) to obtain the SiO_2@NiO adsorbent. The SPE cartridges were made by filling a 5 mL polypropylene syringe with 100 mg of SiO_2@NiO adsorbent. Two polyethylene frits kept the material in place.

2.4. Sample Preparation

The stock standard solution of BaP (500 μg/mL) was prepared in acetonitrile and then stored in the refrigerator at -20℃. Working solutions were freshly prepared daily by appropriate dilution of stock solutions using n-hexane to the desired concentration.

Edible oils (soybean oil, 2 kinds of olive oil, corn germ oil, flaxseed oil, walnut oil, sunflower kernel oil and peanut oil) were purchased from the Taobao internet in China. Edible oil (2.0 g) was accurately weighed and then diluted up to the mark in a 10 mL volumetric flask using n-hexane, which was used for the subsequent SPE.

Two milliliter (2 mL) of the diluted edible oils was directly loaded onto the SiO_2@NiO cartridge, which was preactivated by 2 mL of dichloromethane and 2 mL of n-hexane, at a flow rate of 1 mL/min. The cartridge was then rinsed with 3 mL of n-hexane to eliminate some impurities in oil. BaP was eluted using 5 mL of acetonitrile. Finally, the eluate was concentrated to dryness under nitrogen gas at 40 ℃ and redissolved in 200 μL ACN/H_2O (80/20, v/v) and analyzed by HPLC-DAD.

3. Results and Discussion

3.1. Characterization of SiO_2@NiO

The morphology of the SiO_2 and the SiO_2@NiO was examined by field emission scanning electron microscopy (FE-SEM) and shown in Figure 1. After deposition of NiO, the sizes of SiO_2@NiO almost did not changed. EDS mapping confirmed the presence of nickel element in SiO_2@NiO and not in SiO_2, which indicated that SiO_2@NiO was successfully prepared.

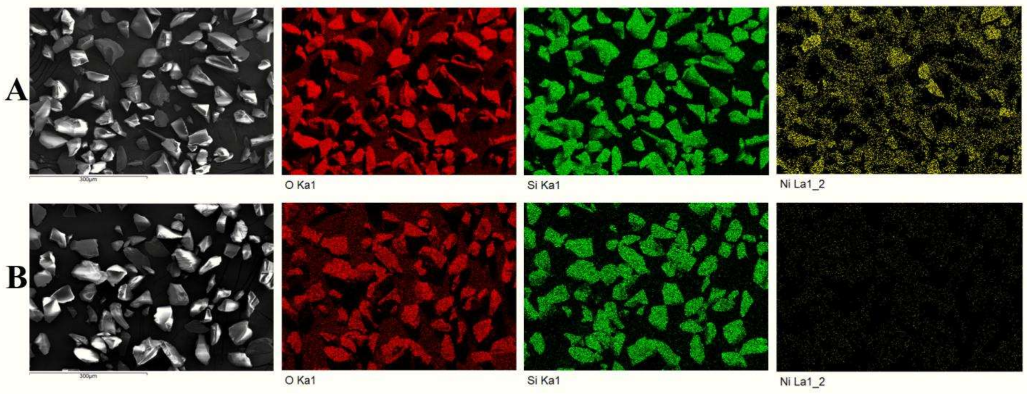

Figure 1. FE-SEM and EDS mapping images of (A) SiO_2@NiO and (B) blank silica (SiO_2).

To evaluate the specific surface area and pore characteristic of the adsorbent before and after deposition of NiO, the nitrogen adsorption analysis was performed. The results presented in Table 1 showed that the specific surface area, pore volume and pore diameter of SiO_2@NiO have no obvious changes, and still remained large specific surface area suitable for use as SPE adsorbent.

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Table 1. Specific surface area, pore volume, and pore size of SiO_2 and SiO_2@NiO.

Adsorbents	Specific Surface Area (m^2/g)	Pore Volume (cm^3/g)	Pore Size (nm)
SiO_2	391.9	0.9	9.8
SiO_2@NiO	396.4	0.9	9.6

3.2. Optimization of SPE

In order to determine the best extraction conditions, the SPE parameters such as the amount of adsorbent, cleaning solvent volume, desorption solvent type and volume were optimized. All the optimization tests were carried out using the blank soybean oil spiked with BaP at a concentration of 50 ng/mL as a representative sample matrix. All the experiments were performed in triplicate. And the recoveries were used for evaluating the extraction efficiency

3.2.1. Influence of Adsorbent Amount

The adsorbent amount was one of the important factors affecting extraction efficiency. Various amounts of adsorbent ranging from 5 to 400 mg (5, 100, 200, 300, 400 mg) were investigated. As shown in Figure 2A, the recovery was maximum when the amount of SiO_2@NiO was 100 mg. Therefore, 100 mg SiO_2@NiO was used to for the further experiments.

3.2.2. Influence of Washing Solvent Volume

Because the polar solvents such as acetone, ethyl acetate, and acetonitrile could occupy the adsorption sites in SiO_2@NiO and could not retain BaP, the nonpolar n-hexane solution can dissolve fat-soluble substances in the edible oil and was used as the loading and cleaning solvent. The volume (2, 3, 4, 5 and 6 mL) of the cleaning solvent was optimized. As shown in Figure 2B, the volume of the cleaning solvent has little effect on the extraction recovery. In order to eliminate enough disturbances and save extraction time, 3 mL n-hexane was finally selected as the suitable washing solvent.

3.2.3. Influence of Desorption Solvent

Not only the adsorption, but also the desorption including the kind of desorption solvent and amount of desorption solvent affected the extraction recovery. Acetone, dichloromethane, methanol, ethanol and acetonitrile was selected as desorption solvent. As shown in Figure 2C, dichloromethane demonstrated the best desorption performance. Therefore, dichloromethane was selected as the desorption solvent. Further, the desorption solvent volume ranging from 2 to 6 mL was optimized. As shown in Figure 2D, 4 mL dichloromethane can elute most BaP (95%). When the volume of dichloromethane was more than 4 mL, the recoveries were stable. In order to completely eluted BaP from SiO_2@NiO, 4.0 mL was selected as the final volume.

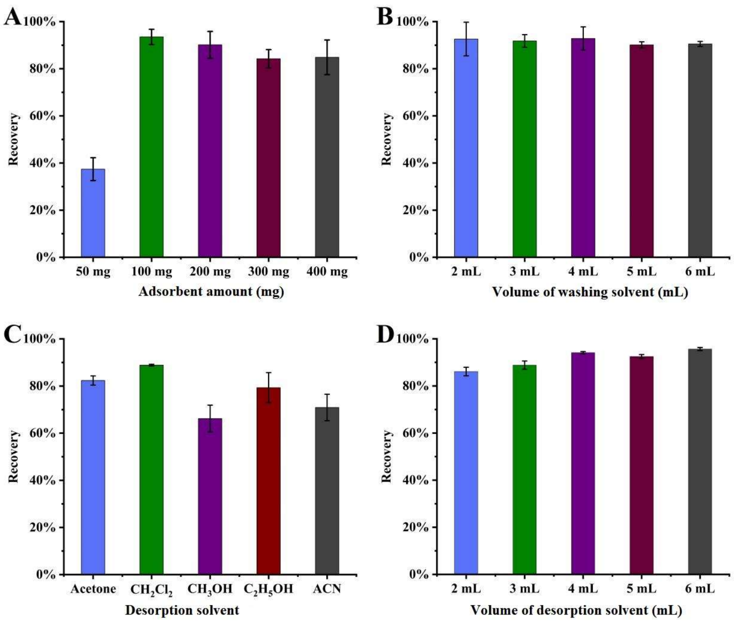

Figure 2. Optimization of SPE conditions. (A) adsorbent amount (B) washing solvent volume (C) desorption solvent type (D) desorption solvent volume.

Based on the above results, adsorbent amount of 100 mg, washing solvent of 3 mL n-hexane and desorption solvent of 4.0 mL dichloromethane were selected as the optimal conditions for SiO_2@NiO SPE.

3.3. Comparison of BaP SPE on SiO_2@NiO and SiO_2

In order to verify the adsorption of nickel oxide coating on SiO_2 for BaP, the extraction efficiency of BaP on SiO_2@NiO and SiO_2 were compared under the same SPE conditions. The results indicated that the extraction recovery of BaP on SiO_2 was only 9.2% (RSD 0.3%), while that on SiO_2@NiO was 87.5% (RSD 0.4%). These results indicate that BaP was captured mainly by the nickel oxide coating on SiO_2@NiO, which can be speculated to the electron donor-acceptor interactions between Ni (Ⅱ) with vacancies and the electron-rich target analyte.

3.4. Reproducibility of SiO_2@NiO

The extraction stability and preparation reproducibility of SiO_2@NiO were investigated by comparing the extraction recoveries of BaP on SiO_2@NiO from the same batch (n=5) and different batches (n=5). As shown in Figure 3, the RSDs of the intra-batch and inter-batch extraction efficiency of SiO_2@NiO were less than 3.2% and 1.5%, indicating that the home-made SiO_2@NiO adsorbents present good reproducibility.

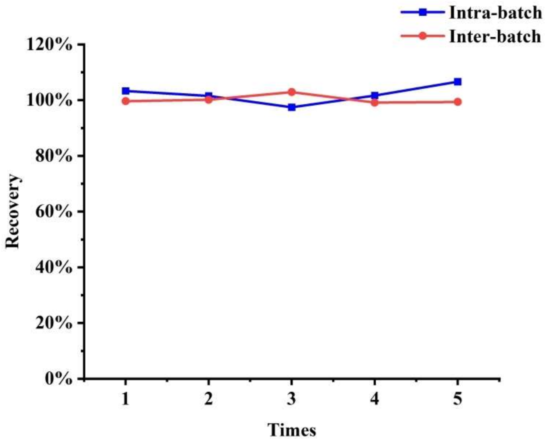

Figure 3. Intra-batch and inter-batch extraction efficiency of SiO_2@NiO adsorbent.

3.5. Method Validation

Figure 4 showed HPLC chromatograms of a blank oil sample of soybean oil (A), as well as the sample spiked BaP at the concentratin of 500 ng/mL (B) and the BaP standard solution with the same concentration (C). Although a cleaner sample solution with less interference can be obtained after SiO_2@NiO SPE, there may be also matrix effect. In this paper, matrix effect was calculated by comparing BaP peak area obtained in the spiked soybean oil with that from standard solutions. The calculated result is 81.7%, indicating that this method still has some matrix effect. Therefore, matrix-matched calibration curve was built to reduce the influence of the matrix for the quantification of samples.

Varying amounts of BaP (12 ng, 30 ng, 60 ng, 150 ng, 300 ng, 750 ng, 1500 ng and 3750 ng) were respectively spiked into 2 g of soybean oil, followed by dilution with n-hexane to prepared matrix-spiked sample solution with concentrations ranging from 6 to 1875 ng/g in soybean oil. Under the optimized SPE conditions, the matrix-matched calibration curve was plotted by the peak area of BaP versus sample concentrations (Table 2). The linear correlation coefficient was calculated to be 0.9999. The limits of detection (LOD) and limits of quantification (LOQ) of the method were calculated at 3 and 10 times the signal-to-noise ratio, respectively. The LOD and LOQ of BaP by the method were 1.3 and 4.4 ng/g, respectively. It was enough for the detection of BaP in edible oils. The spiked samples at three concentration levels (30 ng/g, 150 ng/g and 750 ng/g) were analyzed using the corresponding matrix matched calibration curve. The intra- and inter-day relative standard deviations (RSDs) for the determination were assessed in spiked soybean oil samples at 50 ng/g by 6 determinations in a day for 3 days, respectively. The results showed that the intra- and interday RSDs were less than 3.0 % and 2.6 %, respectively (Table 3). This indicated that the reproducibility of the method was acceptable. The spiked recoveries were varied from 97.4 % to 105.1 %, with RSDs of less than 3.0 % for BaP in spiked soybean oil samples (Table 3). The above results show that the established method has good precision and accuracy, and can be used for the actual sample analysis of BaP in edible oil.

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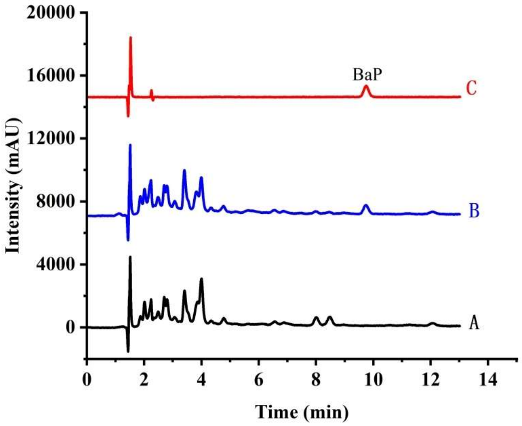

Figure 4. Chromatograms of different sample solutions after SPE. Samples: (A) blank soybean oil sample; (B) blank oil spiked standard sample (C) BaP standard solution.

Table 2. Linearity, limit of detection (LOD), limit of quantification (LOQ) for determination of BaP in oil samples.

Analyte	Linear Range(ng/g)	Regression linear		LOD(ng/g)	LOQ(ng/g)
		Linear equation	R^2		
BaP	6-1875	Y =227.3147X-38.9316	0.9999	1.3	4.4

Table 3. Method recovery and precision (intra-day and inter-day) at three concentrations.

Analyte (BaP)	Concentration	RSD (%)	Recovery (%)
Intra-day (n=6)	Low concentration (30 ng/g)	3.0	97.4
	Medium concentration (150 ng/g)	0.3	100.6
	High concentration (750 ng/g)	1.2	105.1
Inter-day (n=3)	Low concentration (30 ng/g)	2.6	100.1
	Medium concentration (150 ng/g)	0.7	98.9
	High concentration (750 ng/g)	1.1	104.9

3.7. Comparison with Reported Methods

To comprehensively estimate the developed method, it was compared with several reported methods to detect BaP in edible oils, which were presented in Table 4. Firstly, the preparation of adsorbent was simple and did not need complex and rigorous synthesis steps. Secondly, most of the previous methods in Table 4 need a tedious and time consuming liquid extraction before SPE. In the developed method, the oil was only diluted using n-hexane and then directly loaded on SiO_2@NiO for SPE, which simplified the sample processing steps. In addition, the DAD detection equipment used in this method is inexpensive. The method provides satisfactory accuracy and sensitivity. The LOD of BaP in this method was lower than the maximum permissible residual amount of BaP of edible oil in the national standard, which met the daily analysis and detection requirements. All the results indicated that the method using SiO_2@NiO SPE combined with HPLC-DAD for the analysis of BaP in edible oils was simple, economical, effective and accurate.

Table 4. Comparison of this work with other methods.

Adsorbent	Detector	Recoveries (RSDs)	Advantages and drawbacks	Ref.
SPE (SiO_2-OCA)	HPLC-FLD	88.9-122.3% (9.2%)	Low LOD and effective, but tedious and demanding sorbent preparation	[26]
SPE (ProElut C18)	HPLC-DAD-FD	62.6-65.7% (4.9%)	Wide application and practical but low recoveries, complex procedures, and expensive	[33]
MSPE (3D-IL@mGO)	GC-MS	84.4-96.6% (7.9%)	Fast and reusable, but expensive raw materials and tedious production	[34]
SPE (SiO_2@NiO)	HPLC-DAD	97.4-105.1% (3.0%)	Simple, economic adsorbent, effective, stable recoveries, and low cost	This work

3.8. Real Samples Analysis

To verify the practicability of the method, it was applied to the analysis of BaP in 10 oil samples. As shown in Table 5, BaP was detected in 2 unrefined oil and the fried oil. Because unrefined oils were not degummed, deacidified and deodorized, the quality and safety of such oils cannot be guaranteed [35]. The presence of BaP in high-temperature fried oil may be due to some substances in food that are converted into BaP at high temperatures and dissolved in the oil [13]. Therefore, unrefined oil and high-temperature fried oil had the risk of exceeding BaP, which did harm to human health. At the same time, in order to obtain reliable results, 10 ng/g BaP was spiked to the oil sample and then tested to calculate the recoveries of BaP in various oil samples. The recoveries ranged from 87.6% to 111.0% with RSDs in the range of 0.6-10.7%. The results demonstrated that the developed method was applicable for the analysis of BaP from various edible oils.

Table 5. Real concentrations, found concentrations, recoveries, and precisions of BaP in oil samples.

Sample	Soybean oil	Olive oil (1)	Corn oil	Olive oil (2)	Linseed oil	Walnut oil	Sunflower oil	Peanut oil	Unrefined oil (1)	Unrefined oil (2)	Fried oil
Real concentration (ng/g, RSD%)	N.D.	N.D	N.D	N.D	N.D	N.D	N.D	N.D	13.1 (1.8)	8.3 (14.5)	8.6 (7.1)
Found concentration* (ng/g, RSD%)	11.1 (8.4)	10.5 (5.1)	9.9 (10.7)	9.9 (2.0)	10.8 (5.4)	10.9 (1.7)	9.7 (9.3)	9.5 (3.3)	22.9 (0.6)	18.4 (6.4)	17.2 (1.8)
Recovery (%)	111.0	104.9	99.3	99.1	108.9	109.6	97.6	95.0	99.2	103.0	87.6

*The spiked concentration was 10 ng/g. N.D. was not detected.

4. Conclusions

A novel SPE-HPLC-DAD method for the determination of BaP in edible oil was developed using SiO_2@NiO as SPE adsorbent. SiO_2@NiO could selectively capture BaP through electron donor-acceptor interaction. This method exhibited ideal recoveries (97.4-105.1%) and precisions (RSDs 0.3-3.0%). The method possesses great potential for its application in the monitoring of BaP in various edible oils due to its simple sample treatment, easy-to-prepare adsorbent, and low cost.

Author Contributions: Conceptualization, Yinjie Guo and Qiongwei Yu; methodology, Yuejiao Yang. and Yinjie Guo; software, Yinjie Guo and Guanglin Huang; validation, Yinjie Guo; investigation, Yuejiao Yang and Yinjie Guo; resources, Qiongwei Yu; data curation, Yuejiao Yang and Yinjie Guo; writing—original draft preparation, Yuejiao Yang and Qiongwei Yu; writing—review and editing, Guanglin Huang and Qiongwei Yu; visualization,Yuejiao Yang. and Yinjie Guo; supervision, Qiongwei Yu; project administration, Qiongwei Yu. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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