Determination of pesticides and their degradation products in sediment samples by accelerated solvent extraction and solid-phase extraction with high-performance liquid chromatography–high-resolution mass spectrometry

Yan Song1 · Xin Wang1,2 · Ruibao Jia1 · Na Liu1 · Qinghua Zhao1 · Zhangbin Pan1 · Tianxu Zhang1,2 · Shaohua Sun1

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Abstract
A new sensitive and selective methods was developed to quantify different types of pesticides and their degradation products in sediment. The method developed was optimized and modified based on the accelerated solvent extraction, followed by the solid-phase extraction clean-up technique. High-performance liquid chromatography coupled with mass spectrometry was used for analysis. The influence of various parameters on the extraction process was investigated, including the extraction temperature, extraction solvent, purification column and purification solvent, etc. Under the optimal conditions, the relative recoveries of the pesticides and their degradation products ranged from 80 to 106% for spiked blank sediment and environmental sediment samples with relative standard deviations of 1–9%. The method displayed low method detection limits for both sediment matrices and achieved good linearity over the tested range of concentrations. The physical and chemical properties of sediment showed that high content of sediment water content and humic acid would affect the extraction efficiency of sample pretreatment. The method was applied to environmental sediment to quantify pesticide residues in the samples. Based on the instrument and method performance validation results, the developed methods can be applied in environmental pesticide residue analysis, thus providing a scientific method for the detection of sediment samples.

Keywords Sediment · Pesticide degradation products · Accelerated solvent extraction · Solid-phase extraction

Introduction
Sediment is an important part of water ecosystem, which is not only the storage place for nutrients in water bodies, but also for environmental pollutants. Once contaminated, sediments can be further used as chronic sources of pollution. Pesticides and other pollutants in sediments affect not only the survival of benthic organisms, but also the material and energy balance of the whole water [1, 2]. The widespread use of pesticides makes them remain in various environmental matrices. Only a small amount of pesticides applied in farmland show an effect on the target crops, while the rest mostly spread to the soil and the atmosphere, before entering the water body through atmospheric subsidence, surface runoff and other means, and finally accumulate in the sediments [3]. Despite the prohibition on the production and use of certain types of pesticides, contamination of different types of pesticides can still be detected in environmental media such as water, soil and sediment [4, 5]. It is reported that organochlorine pesticides in river sediments of Nigeria are so seriously polluted that both cancer and non-cancer health risk is high in direct contact [6]. Qu et al. [7] found that the total concentration range of organochlorine pesticides in marine sediments in southern Italy was 0.54–99.6 ng g⁻¹. Duodu et al. [8] detected 13 pesticides in the sediments of Brisbane River in Australia with detection frequency more
than 90% and concentration range less than 83.9 ng g\textsuperscript{-1}. The occurrence of different residue levels of pesticides found in these studies may be attributed to their historical application and input from local sources of pesticide use, including agricultural activities, waste water from chemical plants and the use of anti-fouling paints, etc. Compared to the related sediment quality guidelines \cite{9} (the threshold effect concentration of different organochlorine pesticides ranged from 1.90 to 5.28 ng g\textsuperscript{-1}), the probable effect concentration of different organochlorine pesticides ranged from 4.99 to 572 ng g\textsuperscript{-1}), pesticide residues in some locations were higher than the requirements, but the fact that pesticide residues were lower than required in some areas did not mean that they would not pose an ecological risk. As the habitat and nutrient source of aquatic organisms, the accumulation of pesticides in the sediment will affect the water quality and the aquatic ecosystem, endanger the lives of other organisms and disturb the ecological balance \cite{10}.

The hydrophobicity of pesticides makes them less soluble in water and easy to accumulate in sediment. Pesticides in sediments will undergo various biological or abiotic degradation processes, resulting in the decrease of concentration level in the sediment and the increase of transformation products that could be more persistent than the parent compounds. After a 10-year ban on atrazine in Poland, atrazine was not detected in any of the 17 sediment samples collected; however, its degradation products were detected in seven sediment samples \cite{11}. Therefore, it is of great significance to study the detection methods of pesticide residues and their degradation products in sediments for assessing their environmental and health risks as well as strengthening the supervision and management. At present, the commonly used methods for pesticides determination in sediment include but not limited to microwave-assisted extraction \cite{12, 13}, Soxhlet extraction \cite{14}, ultrasonic extraction \cite{15, 16} and accelerated solvent extraction (ASE) \cite{17, 18}. Soxhlet extraction is the most traditional pretreatment method, which have better extraction efficiency and less interference \cite{19}. However, the long extraction time makes the analyte more likely to be resolved by heat \cite{20}. ASE can increase the extraction speed and efficiency by increasing the extraction temperature and pressure. The method is simple, fast, and with little solvent consumption and suitable for the determination of pesticides and their degradation products in sediment.

In addition, sediment matrix is complex and pollutants can accumulate in sediment for a long time, which makes the detection more difficult \cite{21, 22}. Purification procedure improvement is also critical for the precision and accuracy of qualitative and quantitative detection of pollutants in sediment \cite{23}. Solid-phase extraction (SPE) \cite{24} and gel permeation chromatography \cite{25} are frequently used in the analysis of pesticide residues. The SPE method with the features of enrichment and purification can selectively adsorb the target compound to achieve purification effect.

The objective of this study was to develop and apply sensitive extraction methods using ASE and SPE coupled with high-performance liquid chromatography–mass spectrometry for multi-residue analysis of pesticides in sediment. The method was designed for application in the quantification of 13 different pesticides, including three triazine herbicides, one benzothiazole herbicides, one dinitrophenol herbicides, one heterocyclic fungicide, one triazole fungicide, one organophosphorus insecticide, one strobilurin fungicide, one carbamate insecticide, and three degradation products simultaneously. The target analytes were selected based on their popularity through the environmental sediment. The developed method was verified at two different sediments and applied to practical sediment samples. The results proved the higher efficiency, better reproducibility and stability of sample processing.

**Experimental**

**Reagents and chemicals**

Reference standards of ametryne, bentazon, prometryn, prometon, dinoseb, carbendazim, tebuconazole, dimethoate, azoxystrobins, carbofuran, HA(2-hydroxy-4-ethylamino-6-isopropylamino-1,3,5-triazine), DIA (2-chloro-4-ethylamino-6-amino-1,3,5-triazine) and DEA (2-chloro-4-amino-6-isopropylamino-1,3,5-triazine) were supplied from Dr. Ehrenstorfer GmbH (Germany) and ANPEL Laboratory Technologies (Shanghai, China). The most prominent features (log \textit{P} and \textit{pKa}) of the target compounds are presented in Table 1. Standard stock solutions containing 10 mg L\textsuperscript{-1} of all target analytes were prepared in methanol and stored in brown volumetric flasks at 4 °C. Working solutions were prepared by serial dilution of the standard solutions with methanol.

Chromatographic grade solvents (methanol, ethyl acetate, acetonitrile, n-hexane, acetone and dichloromethane) were supplied from Merck (Darmstadt, Germany). ProElut Silica SPE cartridge (1000 mg 6 mL\textsuperscript{-1}), InertSep C\textsubscript{18} SPE (500 mg 6 mL\textsuperscript{-1}) and ProElut Florisil SPE cartridge (1000 mg 6 mL\textsuperscript{-1}) was purchased from Waters (MA, USA). Humic acid (≥ 90%) were obtained from Shanghai Aladdin Bio-Chem Technology (Shanghai, China). Purified water was generated by a Milli-Q Plus purification system (Millipore, Billerica, MA, USA).
Sample collection and preparation

The method was applied to eight environmental sediment samples in Jinan and Jining area. Surface sediment samples were taken in different places within the diameter range of 50 m and were collected with the grab sampler. Mixed and collected in 1 L precleaned brown glass bottles. Triplicate samples were conducted for each sampling site. Samples were transferred to the laboratory and placed on tin foil paper to remove stones, plant debris and other debris. After mixed and air dried, surface sediment samples were sieved through a 20 mesh sieve and stored in 4 °C until analysis. Blank sediment samples were prepared by box-type resistance furnace at 650 °C for 4 h.

Extraction and reconstitution

All samples were extracted using ASE system with 33 mL stainless steel extraction cells. A fiber filter membrane was placed in the bottom of cell prior to loading mixture of 10.0 g of sediment sample and 4.0 g of diatomite particles. The extraction employed methanol as solvent and proceeded at 80 °C, 1500 psi and repeated for 3 cycles. The extracts were collected, and then concentrated to dryness with a rotary evaporator under the water bath heating temperature of 40 °C and vacuum degree of 90 kPa. Finally, 5 mL n-hexane was used for re-dissolution.

A Si SPE cartridge was used for the sediment extract clean-up. The cartridge was activated using 5 mL of n-hexane, and then the concentrated extract was filtered through the cartridge with a flow of approximately 1 drop per second. After sampling, the SPE cartridge was washed by 4 mL of n-hexane, and dried by low vacuum pumping. 6 mL of an acetonitrile–acetone (1:1, v: v) mixture solution was used as elution solvents to recover the target compounds. Finally, the eluents were concentrated and solvent exchange to 2 mL of methanol under a gentle flow of nitrogen, transferred into a sample bottle and stored in refrigerator at 4 °C before analysis.

LC–HRMS conditions

The analysis of target compounds was performed on Dionex Ultimate 3000 Liquid Chromatograph equipped with a Q-Exactive Orbitrap mass spectrometer. An Accucore AQ column (150 × 2.1 mm, 2.6 µm) was used for the sample separation. The mobile phase was water (A) and methanol (B), both containing 0.1% formic acid and 5 mM ammonium formate. The solvent gradient started at 100:0 for A:B (98:2) and B:A (98:2), was applied for 4 min to 80:20 and then increased to 60:40 after 5.5 min; then, it reached 0:100 at 10.5 min and stayed for 4.5 min, and finally to the initial conditions. The flow rate was 0.4 mL min⁻¹. Selected ions and retention times are shown in Table 1. The mass spectrometer used positive and negative electrospray ionization (ESI) under Full MS-ddMS² mode. The full MS scan ranged from 100 to 900 m/z, and the collision gas was N₂. The capillary temperature and probe heater temperature were set at 325 °C and 350 °C, respectively, and the spray voltage was 3000 V.

Experiment on the influence of water content and humic acid

The influence of water content on the extraction efficiency of analytes from sediment samples was assessed under
the following experimental conditions: a certain amount of mixed standard was added to the blank sediment sample to make the concentration of polluted sediment reach 50 µg kg\(^{-1}\), while different amounts of pure water were added to make the water content of sediment sample reach 30%, 50%, 70%, 90% and 110%, respectively.

The effect of different concentrations of humic acid on the extraction efficiency of target compounds was investigated under the following experimental conditions: a certain amount of mixed standard solution was added to the blank sediment sample to prepare polluted sediment sample with the concentration of 50 µg kg\(^{-1}\). 10 mL 0.03 mol L\(^{-1}\) NaCl solution and an amount of humic acid were added into 10 g polluted sediment samples, so that the humic acid concentration in the water that interacted with the polluted sediment was 2, 5, 10 and 20 mg L\(^{-1}\), respectively. After 48 h to continuous oscillation at room temperature and 24 h to static equilibrium, the samples were centrifuged and dried for enrichment and extraction.

**Results and discussion**

**Optimization of ASE method**

The extraction temperature, static extraction time and cycle number of ASE has a direct impact on the extraction efficiency of pesticides and their degradation products. In order to avoid the singularity and opportunity of single-factor experiments, orthogonal experiments were designed to evaluate the recovery index. The level of each factor is: extraction temperature: 80, 100, 120 °C; static extraction time: 5, 10, 15 min; cycle time: 1, 2, 3 times. The orthogonal experiment design scheme and results of ASE method are shown in Table S1 and S2. It can be seen from the results of orthogonal experiment and data processing that the primary and secondary relationship affecting the extraction rate of the target compound is that static extraction time > cycle number > extraction temperature. The optimal extraction conditions obtained by orthogonal experiment were extraction temperature 80 °C, static extraction time 5 min, 3 cycle times.

The effects of methanol, n-hexane, ethyl acetate, dichloromethane, and acetonitrile on the extraction of the target compounds were compared. The results are shown in Fig. 1. Ethyl acetate, n-hexane and dichloromethane as extraction solvents had poor extraction effects on dimethoate and bentazon, the recoveries of dimethoate and bentazon with ethyl acetate, n-hexane and dichloromethane were 102%, 55% and 85%, 89% and 0%, while methanol and acetonitrile could extract target compounds effectively. Acetonitrile is concentrated at relatively high temperatures using rotary evaporators, and the recovery of methanol to target compound is above 90%, therefore, methanol is preferred as extraction solvent.

**Optimization of SPE purification method**

The organic components in the sediment are purified by SPE. According to the characteristics of pesticides and their degradation products, Si, C\(_{18}\) and Florisil columns were optimized and compared. Moreover, the loading volume, washing solution volume, and purification solution volume also directly affect the purification efficiency. The optimal purification conditions were optimized by orthogonal design, and the recovery rate was used as the evaluation index. The level of each factor is purification column: Si, C\(_{18}\) and Florisil column; loading volume: 2, 5 and 10 mL; washing solution volume: 2, 4 and 6 mL; purification solution volume: 2, 4 and 6 mL. The orthogonal experiment design scheme and results of SPE purification method are shown in Table S3 and S4. Comprehensive analysis and results indicate that the primary and secondary influence degree of each factor on SPE purification method was purification solution volume > loading volume > washing solution volume > purification column. The optimal purification conditions obtained by orthogonal experiment were Si purification column, loading volume 5 mL, washing solution volume 4 mL, and purification solution volume 6 mL. Due to the generally low recovery obtained by orthogonal experiments, the choice of purification solvent is critical.

The purification solvent was first compared to the results obtained with ethyl acetate, dichloromethane and acetone. As shown in Fig. 2, the extraction efficiency of acetone was significantly better than that of ethyl acetate and dichloromethane, but the recovery of acetone to HA was relatively low. Considering that ethyl acetate, dichloromethane, and acetone were not effective as purification solutions, the washing solution was retained for analysis, but no loss of compounds was detected. Thus, the compound may remain
on the purification column without being eluted. Due to the low solubility of methanol and acetonitrile as washing solution, the SPE column purified by ethyl acetate was eluted again with methanol and acetonitrile to evaluate the recovery effect of methanol and acetonitrile as purification solvents. Both methanol and acetonitrile had good recovery effect on HA. The recovery rates of HA by methanol and acetoni-
trile were 86% and 93%, respectively. Therefore, the mixed solution was selected for purification. As concentration of methanol with nitrogen blowing was relatively time-con-
suming, acetonitrile and acetone mixture was selected as the purification solvent. Then, the ratio of the mixed solvent is further optimized, so was the proportion of mixed solvent.

The purification effect of acetonitrile–acetone mixture with volume ratio of 1:5, 1:2, 1:1, 2:1, 2:1 and 5:1 was inves-
tigated, respectively. As shown in Fig. 3, the recovery effect of volume ratio at 1:1 is relatively balanced for the target compounds, generally reaching more than 80%. Therefore, acetonitrile-acetone (1:1, v: v) mixture solution was used as SPE purification solvent in the study.

**Influence of sediment conditions on pretreatment methods**

**Effect of water content**

The water content of sediment is an influential factor for extraction, since it simultaneously affects the volatilization time and rate of compounds in sediment [26]. The influence of water content on the extraction efficiency of analytes from sediment samples was assessed. As seen in Fig. 4, C0 is the amount of pesticides at dried sediment samples, and C is the amount of pesticides at different water content. The recoveries of target compound decreased with increased water content between 30% and 110%. This outcome can likely be attributed to the long drying time of the high-water content sediment, resulting in a high loss of organic matter volati-
lization, even to 27% for the highest loss of organic matter. However, the volatilization rate of target compounds with water content in the range of 90–110% is slower than that of 30–90%.

**Effects of humic acid**

The environmental sediment contains a large amount of organic matter, which will be decomposed by microorganism and produce humic acid. The presence of humic acid may interfere with the extraction of pesticides by the treatment system. The effect of different concentrations of humic acid on the extraction efficiency of target compounds was investigated. The solubility organic carbon of humic acid solution (2 mg L−1) was 1.01 mg L−1, SUVA254 was 7.82 L (mg m)−1 and humification index (HIX) was 3.66. The larger SUVA254, the higher the aromaticity of humic acid and the higher HIX value indicated a higher degree of humification and a stable humic acid [27, 28]. As seen in
Fig. 5, C0 is the pesticide content measured without humic acid, and C is the pesticide content measured with different humic acid concentration. The adsorption efficiency has no obvious change when the concentration of humic acid was 2 mg L⁻¹, whereas the concentration of humic acid higher than 2 mg L⁻¹ causes a significant decrease in adsorption. The results show that high concentration of humic acid may interfere the extraction system.

Validation of the analytical methodology

The results of the method validation for blank sediment and environmental sediment are shown in Table 2. At each spiked level, the mean recoveries ranged from 81 to 106% for blank sediment, and 80–102% for environmental sediment. It was observed that the calculated concentrations were comparable to the actual concentrations in both matrices. The method exhibited good repeatability with relative standard deviations (RSD) generally lower than 10%. Matrix effects were investigated in environmental sediment sample at a spiked level of 50 µg kg⁻¹. The results showed that the matrix effect for target analytes ranged from −17 to 11%. Matrix components did not produce a significant impact on the detection signal responses of target analytes. Performance analyses show that the optimized method meet the relevant detection requirements.

Method performance parameters including linear ranges, linearity and the method detection limits (MDLs) and the method quantitation limits (MQLs) were obtained by the determination of spiked known amount standards into blank sediment. The method performances were summarized in Table 3. Analysis of the target compounds showed acceptable linearity in the measured concentration ranges from 1.0 to 200 µg kg⁻¹ with correlation coefficients over 0.9915. The MDLs and MQLs for the target compounds in blank sediment were 0.0020−0.37 µg kg⁻¹ and 0.0080−1.5 µg kg⁻¹, respectively. The MDLs was slightly lower than previous studies 0.0060−1.0 µg kg⁻¹ by ASE–SPE–GC/MS and 0.10−57.1 µg kg⁻¹ by ultrasound assisted extraction (UAE)/ASE–SPE–LC–MS/MS [29–31].

Determination of pesticides and their degradation products in sediment samples

The developed method was applied to environmental sediments collected at different water to quantify thirteen target...
Determination of pesticides and their degradation products in sediment samples by accelerated.

The pH, conductivity, organic matter content and water content of the collected sediments were 7.50–8.10, 638–1302 μs cm⁻¹, 1.97–8.87% and 13.9–38.2%, respectively (Table 4). The mean concentrations of the pesticide residues are presented in Table 5. Different concentrations of ametryne, prometryn, prometon, tebuconazole and DEA were detected in 8 sediment samples. And DEA was the most frequently detected pesticide degradation product, with the concentration range 0.11–8.5 μg kg⁻¹.

### Conclusions

An ASE–SPE purification method for the determination of 13 pesticides and their degradation products in sediment was established. Critical factors affecting the extraction efficiency including accelerated solvent extraction temperature, static extraction time, cycle times, SPE purification column, sample loading volume, eluent volume, and eluent volume were systematically studied. In addition, research analysis shows that high content moisture and humic acid of sediment affect the extraction efficiency of sample pretreatment. This

| Analyte     | Linear range, µg kg⁻¹ | $R^2$  | MDLs, µg kg⁻¹ | MQLs, µg kg⁻¹ |
|-------------|------------------------|--------|---------------|---------------|
| Ametryne    | 1–100                  | 0.9968 | 0.0020        | 0.0080        |
| Bentazon    | 1–100                  | 0.9918 | 0.0020        | 0.0080        |
| Prometon    | 1–100                  | 0.9927 | 0.012         | 0.048         |
| Prometryn   | 1–100                  | 0.9927 | 0.042         | 0.17          |
| Dinoseb     | 10–200                 | 0.9936 | 0.060         | 0.24          |
| Carbendazim | 1–100                  | 0.9979 | 0.011         | 0.044         |
| Tebuconazole| 1–100                  | 0.9987 | 0.0050        | 0.020         |
| Dimethoate  | 10–200                 | 0.9973 | 0.027         | 0.11          |
| Azoxystrobin| 1–100                  | 0.9948 | 0.045         | 0.18          |
| Carbofuran  | 1–100                  | 0.9967 | 0.052         | 0.21          |
| HA          | 1–100                  | 0.9988 | 0.056         | 0.22          |
| DIA         | 1–100                  | 0.9915 | 0.37          | 1.5           |
| DEA         | 1–100                  | 0.9996 | 0.046         | 0.18          |

### Table 4 Physical and chemical properties of the eight environmental sediment samples

| Analyte         | Xiaoqing river sediments | Yellow river sediments | Nansi lake sediments |
|-----------------|--------------------------|------------------------|----------------------|
|                 | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 |
| pH              | 7.50 | 7.60 | 7.70 | 7.90 | 8.10 | 7.80 | 8.00 | 7.94 |
| EC (μs/cm)      | 839 | 733 | 953 | 779 | 1302 | 1013 | 1107 | 638 |
| Organic mass (%)| 8.87 | 4.86 | 3.52 | 2.84 | 2.23 | 1.97 | 2.59 | 2.53 |
| Water content (%)| 33.3 | 37.4 | 32.4 | 38.2 | 15.8 | 13.9 | 14.2 | 28.9 |

### Table 5 Concentration of target analytes in eight environmental sediment samples (µg kg⁻¹, $n=3$)

| Analyte     | Xiaoqing river sediments | Yellow river sediments | Nansi lake sediments |
|-------------|--------------------------|------------------------|----------------------|
|              | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 |
| Ametryne    | ND | ND | 8.6±1 | ND | 1.48±5 | 3.72±2 | 4.84±6 | ND^a |
| Bentazon    | ND | ND | ND | ND | 18.16±2 | ND | ND | 0.04±2 |
| Prometon    | ND | ND | 3.3±14 | 2.7±6 | ND | ND | ND | ND^a |
| Prometryn   | ND | ND | 3.8±9 | 3.0±7 | ND | ND | ND | ND^a |
| Dinoseb     | ND | ND | ND | ND | 17.02±1 | 14.7±4 | 30.62±4 | ND |
| Carbendazim | ND | ND | ND | ND | ND | ND | ND | ND |
| Tebuconazole| 0.72±11 | 0.85±7 | 4.7±9 | 1.48±5 | 0.78±6 | 0.94±8 | ND^a |
| Dimethoate  | ND | ND | ND | ND | ND | ND | ND | ND^a |
| Azoxystrobin| ND | ND | ND | ND | ND | ND | ND | ND^a |
| Carbofuran  | ND | ND | ND | ND | ND | ND | ND | ND |
| HA          | ND | ND | ND | ND | ND | ND | ND | ND |
| DIA         | ND | ND | ND | ND | ND | ND | ND | ND |
| DEA         | 0.11±2 | 0.26±6 | 8.5±4 | 7.3±2 | ND | ND | ND | 0.18±3 |

^aND, not detected
method has been successfully used to determine pesticides and their degradation products in environmental sediments. Overall, good adsorption and purification capabilities, high stability and repeatability of the developed method have been proven in determination of complex environmental sediments.

**Supporting Information**

This material is available free of charge on the Web at http://www.jsac.or.jp/analsci/.

**Supplementary Information** The online version contains supplementary material available at https://doi.org/10.1007/s44211-022-00165-9.

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**Declarations**

**Conflict of interest** No competing interests exits in this submitted manuscript, and all the authors listed have approved the manuscript for publication. The manuscript describes original research that has not been published previously.

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