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Synthesis and application of molecularly imprinted polymer on selective solid-phase extraction for the determination of monosulfuron residue in soil

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Abstract

A novel sample clean-up procedure using molecularly imprinted polymer as the solid-phase extraction material for the determination of monosulfuron residue in soil samples has been developed. The molecularly imprinted polymer (MIP) was synthesized by non-covalent method with monosulfuron as the template. The selectivity and affinity of the MIP was evaluated by equilibrium adsorption and HPLC experiments, which demonstrated that the MIP has specific affinity for the template. The template-MIP interaction was studied by investigating the influence of different mobile phases on the retention of the template, which provided basic knowledge for the selection of the washing and elution solutions in the molecularly imprinted solid-phase extraction (MISPE) process. The study indicated that polar organic solvents with hydrogen bonding abilities have stronger eluting strength for the monosulfuron. After the MISPE procedure, a clean baseline was obtained in the HPLC quantification analysis. The recoveries of the method using the combination of MISPE and HPLC were above 93% and the R.S.D. was less than 3.2% in the soil sample determinations. Low detection limit (0.08 $\mu g\, g^{-1}$, when defined as 3 times of the noise) was also obtained in the method evaluation study.

Keywords: Molecularly imprinted polymer; Solid-phase extraction; Monosulfuron; Residue determination

1. Introduction

Monosulfuron {*N*-[(4′-methyl) pyrimidin-2′-yl]-2-nitrophenylsulfonyl urea} is a new sulfonylurea herbicide with good herbicidal activity and low toxicity for mammals developed by the National Pesticide Engineering Research Center in Tianjin, China. As a pesticide to be used in field, its persistence in soil has to be evaluated. A method for the trace determination of monosulfuron in soil is required due to the low application rate and quick dissipation of the sulfonylurea herbicides in crop environment. This is a challenging work due to the co-extraction of soil impurities when the conditions that are favorable for the monosulfuron extraction was used because the polarity of monosulfuron is similar to the humic acids in soil. The previous determination of the monosulfuron residue in soil involved complicated and labor intensive steps that included traditional liquid–liquid and solid-phase extraction and then HPLC analysis [1,2]. Development of a more convenient process for the sample clean-up is necessary.

Molecular imprinting is a technology to make receptor-like polymers based on the molecular recognition theory and was rapidly developed in recent years [3–6]. Molecularly imprinted polymers (MIPs) have shown advantages such as pre-determined selectivity and chemical stability in many analytical areas [7–12]. Applications of MIPs in the solid-phase extraction (SPE) [13–21] demonstrated that MIPs have

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great potential for the complex sample clean-up especially when the analyte and impurities have similar properties. To develop a more efficient method for the determination of the monosulfuron residue in soil and evaluate the feasibility of using MIP for the SPE of monosulfuron, MIP used for the sample clean-up was synthesized. Washing and elution steps in the molecularly imprinted SPE/(MISPE) were optimized after the MIP properties were evaluated. A reversed-phase HPLC procedure was used for the analyte quantification after the solid-phase extraction process.

Soil samples containing spiked monosulfuron were analyzed with the MISPE–HPLC method. Good recoveries and the R.S.D. values have shown the procedure provides reliable results. The MIP synthesis, evaluation and MISPE studies in the research are presented in this paper.

2. Experimental

2.1. Materials and chemicals

Monosulfuron, chlorsulfuron, imidacloprid and NK No. 94827 were provided by the National Pesticide Engineering Research Center (Tianjin, China). Methacrylic acid (MAA) was purchased from Donghuan United Chemicals (Beijing, China) and distilled under vacuum before use. Ethylene dimethacrylate (EDMA) was from Anli Chemical Co. (Suzhou, China). AIBN [2,2′-azobis(2-methylpropionitrile)] was from the Special Reagent Labs. of affiliated school of Nankai University (Tianjin, China). Water (pH 3) was prepared by adjusting the pH of the de-ionized water with H_3PO_4. Soil samples (from Shandong and Sichuan provinces, China) were air-dried at room temperature in flat stainless steel trays, homogenized, and then sieved with a 40 mesh screen before use.

2.2. Molecularly imprinted polymer preparation

Non-covalent method with monosulfuron as the template was employed in the molecularly imprinted polymer synthesis. In the synthesis, monosulfuron (1.6 mmol), functional monomer (MAA, 7.8 mmol), cross-linker (EDMA, 39 mmol) and initiating agent (AIBN, 0.6 mmol) were dissolved in the porogen (dimethylformamide, 17 mL). The molar ratio of template/monomer/cross-linker was 1:5:25. The solution was transferred into a glass tube and deoxygenated with N_2 stream. The glass tube was flame sealed and irradiated with 366 nm UV light at ambient temperature. After 24 h, the resulting bulk polymer was ground and wet sieved in water. Polymer particles with sizes less than 36 μm were collected and fine particles were removed by repetitive sedimentations with acetone. The polymer particles were washed with 0.2 mol L^{-1} ammonia in water–MeOH (1:1 (v/v)) to extract the monosulfuron from the polymer. Non-imprinted polymer was synthesized with the same polymerization conditions except no template was added.

2.3. MIP binding property and selectivity evaluation

Equilibrium adsorption experiments were used to evaluate the binding affinity of the imprinted polymer. Polymer particles (30 mg) were mixed with 5.0 mL acetonitrile containing monosulfuron at various concentrations (from 0.03 to 0.6 mmol L^{-1}). The mixtures were incubated at ambient temperature with continuous shaking for 8 h. After incubation, the mixtures were centrifuged. The concentration of monosulfuron in the supernatant solution was determined by measuring the UV absorption at 237 nm using a UV–vis spectrometer (Shimadzu UV–vis 240). The amount of monosulfuron bound to the polymers, *B*, was calculated by subtracting the amount of unbound monosulfuron from the amount of monosulfuron added to the mixture. Data from triplicate measurements were averaged.

Chromatographic separation of the monosulfuron and its structural analogs was performed to study the selectivity of the MIP. Chromatographic experiments were carried out with an Agilent 1100 HPLC system and monitored at 237 nm. MIP particles slurry-packed in a 150 mm × 4.6 mm stainless steel column were used as the stationary phase. The mobile phase used for the analysis was aqueous buffer (0.1 mol L^{-1} HOAc–NaOAc, pH 3.6)–MeOH (4:6 (v/v)) with a flow rate of 1 mL min^{-1}. The retention factor was calculated as $k = (t - t_0)/t_0$ where t is the retention time of the analyte and t_0 is the retention time of acetone which was used as the void marker. The selectivity factor α was calculated as $\alpha = k_m/k_x$ where k_m and k_x are the retention factors of the monosulfuron and its analogs, respectively.

The aqueous buffer–MeOH solutions were used in the study of the effect of mobile phase on the retention of monosulfuron on the MIP column. Aqueous buffers (with different pH) were prepared according to the Handbook of Biochemistry and Molecular Biology (0.1 mol L^{-1} citrate–0.2 mol L^{-1} Na_2HPO_4 for pH 3; 0.1 mol L^{-1} HOAc–NaOAc for pH 4 to pH 5; 0.07 mol L^{-1} Na_2HPO_4–0.07 mol L^{-1} KH_2PO_4 for pH 7 buffers, respectively) [22].

2.4. Sample and SPE cartridge preparation

The background solutions for the MISPE study which contained impurity substances from soil were prepared according to the following procedure. Ten grams of soil sample was mixed with 30 mL of extraction solution (0.2 mol L^{-1} ammonia in water–MeOH, 1:1 (v/v)). After being sonicated for 45 min, the mixture was shaken with a horizontal shaker for 1 h and then filtered under vacuum. The filtrate was dried in a rotary evaporator at 60 °C. The background solutions were then made by dissolving the residue in 0.5 mL acetonitrile (MeCN). The test solutions for the MISPE evaluation were prepared by spiking monosulfuron into the background solution.

Two types of MISPE cartridges were used in the SPE studies. One cartridge was a 5 mL polypropylene tube with

250 mg MIP by dry packing and secured by frits (cartridge 1). The second cartridge was a 50 mm × 4.6 mm stainless steel HPLC column packed with 400 mg MIP (cartridge 2).

2.5. Evaluation of the washing solution for the solid-phase extraction with MISPE cartridge 1

MISPE cartridge 1 was used to evaluate different washing and elution solutions. The cartridge was first conditioned with 10 mL MeCN and then a 0.5 mL volume of MeCN containing monosulfuron (50 μg mL^{-1}) was loaded at a flow rate of 0.2 mL min^{-1}. The cartridge was washed with 3 mL of washing solution and then 5 mL of elution solvent with a flow rate of 0.5 mL min^{-1}. The eluates were collected and dried under an air stream when organic washing solvents were used or with a rotary evaporator when water (pH 3.0)–MeOH (7:3 (v/v)) was the washing solution. The residue was dissolved into 100 μL of water/(pH 3.0)–MeCN (4:6 (v/v)) for the HPLC analysis to determine the amount of monosulfuron recovered from the loading, washing and elution steps.

2.6. Solid-phase extraction with MISPE cartridge 2

MISPE cartridge 2 was used to optimize the SPE conditions and used for the sample clean-up process in the developed analytical method. The SPE experiment was performed with a liquid chromatographic system consisting of an Agilent 1100 HPLC, a Shimadzu LC-4A pump and a Rheodyne 7040 valve (Fig. 1). For the MISPE process, SPE cartridge 2 was conditioned with washing solution (solution A) from pump A. After the sample (100 μL) was injected, solution A was pumped into the cartridge to elute the impurities. Then the valve was switched. The column was back flashed with elution solution (solution B) from pump B. The eluted fraction was collected and dried with a gentle air stream. The residues were dissolved in 100 μL of water/(pH 3.0)–MeCN (4:6 (v/v)) and analyzed by reversed-phase analytical HPLC.

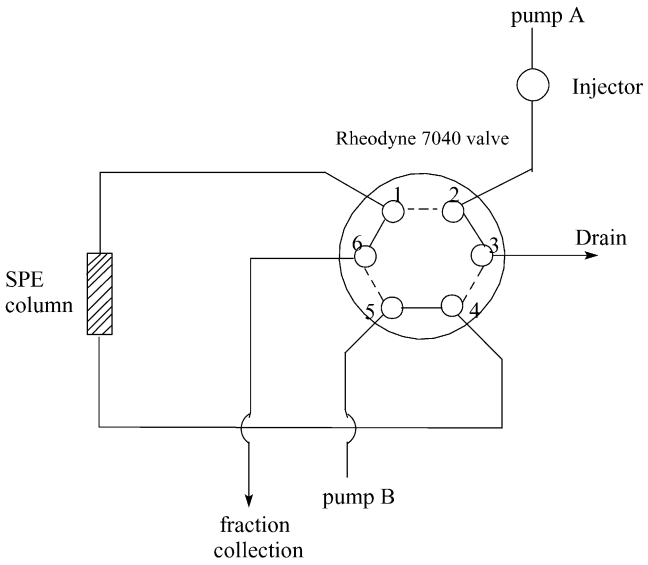

Fig. 1. Instrument setup for the solid-phase extraction with MISPE cartridge 2. In the setup, pump A was from the Shimadzu 4A HPLC instrument while pump B was from the Agilent 1100 HPLC instrument.

2.7. Soil sample preparation and solid-phase extraction

Dry soil sample (10 g) was weighed and mixed with 10.0 mL of acetonitrile solution containing monosulfuron. The mixture was sonicated for 1 h and then left standing for 12 h. The acetonitrile was then evaporated in a ventilation cabinet at room temperature to obtain the monosulfuron spiked dry soil samples.

The spiked dry soil sample was mixed with 30 mL of 0.2 mol L^{-1} ammonia in water–MeOH (1:1 (v/v)) solution to extract monosulfuron. The mixtures were sonicated for 45 min and then shaken for 1 h in a horizontal shaker. The extracts were filtered under vacuum. The filtrate was dried in a rotary evaporator to nearly dryness at 60 °C. The remaining solvent was evaporated by an air stream. Acetonitrile (0.5 mL) was then added to dissolve the residue for the SPE process.

For the SPE process, the cartridge 2 was conditioned with water (pH 3.0)–MeOH (7:3 (v/v)). After loading of 100 μL sample solution, the MISPE column was washed with water (pH 3.0)–MeOH (7:3 (v/v)) for 8 min. Then the column was back flashed with solution B (0.5% trifluoroacetic acid in MeOH) to elute the monosulfuron. The fraction was collected and evaporated with an air stream to dryness. The residue was dissolved with 100 μL of water (pH 3.0)–MeCN (4:6 (v/v)) and analyzed with reversed-phase analytical HPLC.

2.8. Reversed-phase HPLC analysis for monosulfuron quantification

Reversed-phase HPLC analysis was used for the quantification of the monosulfuron after the solid-phase extraction. The instrument was an Agilent 1100 system consisting of a quaternary pump and multiple wavelength UV–vis detector. A 150 × 4.6 mm Zorbax C_{18} column was employed for the analysis. The detection wavelength was 237 nm. The mobile phase was water (pH 3.0)–MeOH (55:45 (v/v)) with a flow rate of 1.0 mL min^{-1}.

3. Results and discussion

3.1. MIP preparation and evaluation

MAA, which may form hydrogen bonding with monosulfuron, was selected as the functional monomer in the MIP preparation. Because proper template concentration in the polymerization mixture is the requirement for creating enough binding sites in the MIP while monosulfuron has very poor solubility in non-polar and some polar organic solvents

Table 1
Solubility of monosulfuron in different organic solvents at 25 °C [23]

Solvent	Solubility (mg L^{-1})
DMF	32885.39
Chloroform	918.65
Dichloromethane	1698.48
Acetonitrile	1050.26
Acetone	1803.52
Methanol	223.22

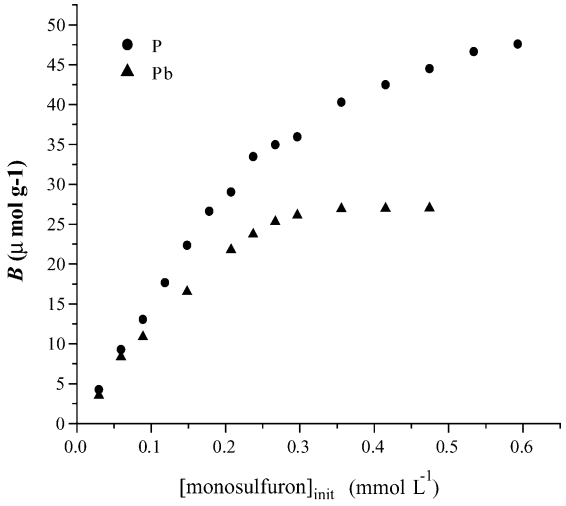

Fig. 2. Binding isotherm of monosulfuron on imprinted and non-imprinted polymers. P: molecularly imprinted polymer, Pb: non-imprinted polymer; $[monosulfuron]_{init}$ represents the initial concentration of monosulfuron.

which are generally used in molecular imprinting (Table 1), dimethylformamide (DMF) in which monosulfuron can be dissolved in a desired concentration was chosen as porogen.

The binding affinity of the resulting MIP was evaluated by equilibrium adsorption experiment. The results showed that the monosulfuron imprinted polymer has higher affinity for the template than the non-imprinted polymer (Fig. 2). The Scatchard analysis (data omitted) also showed that imprinted polymer has higher binding association constants and more apparent binding sites than the non-imprinted polymer, which may demonstrate the binding affinity of the MIP is from the specific bind sites by the imprinting effect.

The selectivity of the MIP was evaluated by measuring its ability to resolve structural analogs in the HPLC process. The MIP particles were slurry packed into a 150 mm × 4.6 mm stainless steel column and used as the stationary phase. Three

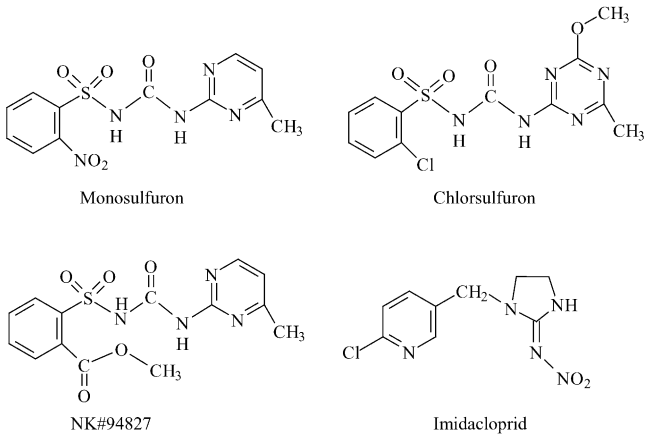

Fig. 3. Chemical structures of the compounds (sulfonylurea herbicides and imidacloprid) used in the selectivity evaluation.

sulfonylurea herbicides and one pesticide with a smaller molecular size (imidacloprid) were used for the study (Fig. 3). The selectivity factors obtained from the experiment are listed in Table 2.

The selectivity experiment demonstrates that the MIP is able to recognize the structural differences between the template and its analogs, including different size of the adjacent substituent on the benzene ring in the sulfonylurea structure (monosulfuron versus NK No. 94827). The affinity and selectivity evaluation indicated that the imprinted polymer is a potential SPE separation material having selectivity for the monosulfuron.

3.2. Study of the interaction properties of monosulfuron with the MIP

To obtain information about the interaction of monosulfuron with the MIP, the influence of the mobile phases on the retention of the monosulfuron was studied. The HPLC analysis was performed on a slurry packed MIP column (150 mm × 4.6 mm). In the first set of experiments, the pH of the mobile phases (aqueous buffer–MeOH) was varied. The buffer pH was changed from 2.6 to 6.0 while the ratio of aqueous buffer–MeOH was fixed as 4:6. Another set of experiments was carried out to study the influence of buffer concentration in the mobile phase on the retention of the monosulfuron. In the experiment, the mobile phases were (0.1 mol L^{-1} HOAc–NaOAc, pH 3.6)–MeOH and the con-

Table 2
The retention and selectivity factors for pesticides on the MIP column[a]

Monosulfuron	Retention factors (k)			Selectivity factors (α)[b]		
	Chlorsulfuron	NK No. 94827	Imidacloprid	k_M/k_C	k_M/k_N	k_M/k_I
8.4	6.7	5.1	4.0	1.3	1.7	2.1

[a] HPLC column: a 150 mm × 4.6 mm stainless steel column packed with MIP. Mobile phase: (0.1 mol L^{-1} HOAc–NaOAc, pH 3.6)–MeOH (4/6 (v/v)). Flow rate: 1 mL min^{-1}.

[b] Selectivity factors for monosulfuron versus other compounds. k_M, k_C, k_N, k_I are the retention factors of monosulfuron, chlorsulfuron, NK No. 94827 and imidacloprid, respectively.

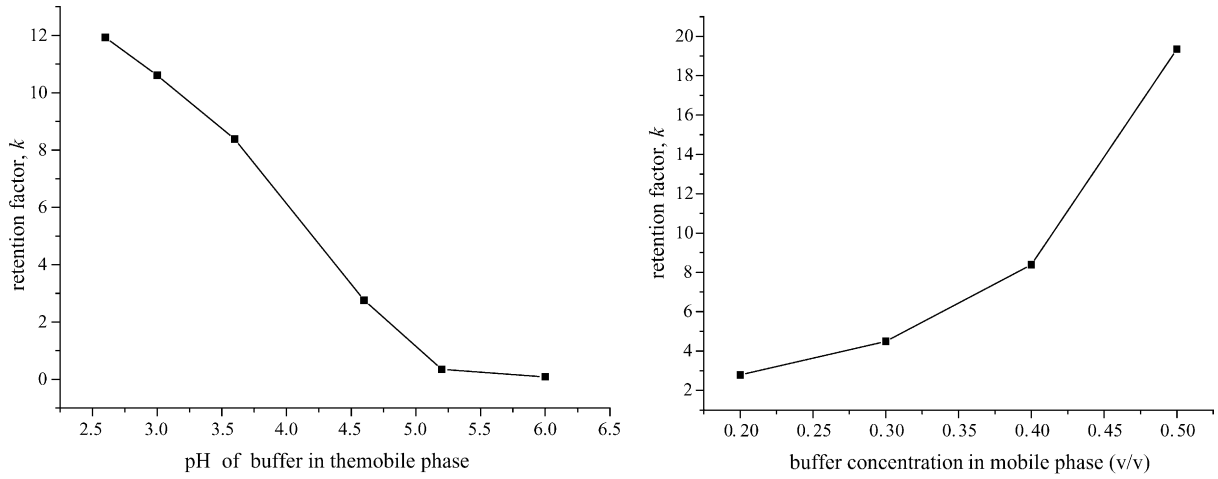

Fig. 4. The influence of mobile phase on the retention factor of monosulfuron. In the HPLC analysis, the column was a stainless steel column (150 mm × 4.6 mm) with slurry packed MIP. Mobile phase: (a) aqueous buffer–MeOH (4:6) while pH of aqueous was varied from 2.5 to 6.0; (b) (0.1 mol L^{-1} HOAc–NaOAc, pH 3.6)–MeOH while buffer concentration was changed from 0.2 to 0.5 in volume.

centrations of the buffer in mobile phases were varied from 0.2 to 0.5 in volume.

The experimental results (Fig. 4) showed that a decrease of buffer pH or an increase of the aqueous proportion in the mobile phase resulted in higher retention of monosulfuron on the MIP column. Because monosulfuron is an acidic compound with an estimated pK_a value in the range of 3–4, more monosulfuron molecules exist in a non-ionized form under lower pH conditions. The results of the experiments suggested that a reversed-phase mode-like retention behavior existed on the MIP column under these test conditions.

To evaluate the ability of MIP to separate the soil impurities from the analyte, the background solutions spiked with monosulfuron were analyzed on the MIP column (150 mm × 4.6 mm). Good separation was obtained using the buffer (0.1 mol L^{-1} citric acid–0.2 mol L^{-1} Na_2HPO_4, pH 3.0)–MeOH (4:6 (v/v)) as the mobile phase (Fig. 5).

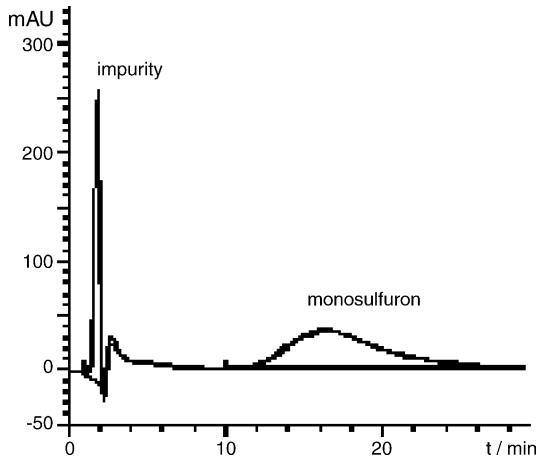

Fig. 5. Separation of monosulfuron from soil impurities on the MIP column. Column: a 150 mm × 4.6 mm stainless steel column packed with MIP. Mobile phase: buffer (0.1 mol L^{-1} citric acid–0.2 mol L^{-1} Na_2HPO_4, pH 3.0)–MeOH (4:6 (v/v)). Flow rate: 1.0 mL min^{-1}.

3.3. Optimization of the washing and elution conditions for the MISPE procedure

Because an off-line procedure using SPE cartridge 1 (polypropylene tube packed with MIP) is more convenient for changing solvent with various properties, it was used for washing and elution solvents evaluation at the first stage. The flow of the solution through the SPE column was driven by vacuum. After the cartridge was conditioned with 10 mL of acetonitrile, 0.5 mL of monosulfuron in acetonitrile (50 μg mL^{-1}) or the background solution was loaded. Different washing solutions were applied to evaluate their ability of eluting the template and soil impurities from the column. The eluate from the cartridge in the washing steps was collected and dried with an air stream. The recovery of monosulfuron in the washing steps was measured by the reversed-phase HPLC. For the solutions evaluated in the experiment, MeOH had the strongest elution strength for the template followed by the less polar solvent $CHCl_3$ (Table 3). This result indicated that a polar organic solvent with hydrogen bonding ability is required for eluting the template from the column with higher efficiency, which suggested that both

Table 3
Recovery[a] of monosulfuron (25 μg) with different washing solutions[b] (n = 3)

Washing solutions	Recovery, % (R.S.D., %)
CH_3OH	89.3 (3.70)
CH_3CN	56.3 (1.13)
$CHCl_3$	62.2 (3.09)
CH_3COCH_3	52.4 (3.89)
Water (pH 3.0)–CH_3OH (7:3 (v/v))	46.6 (2.79)

[a] The recovery was calculated by the amount of monosulfuron eluted at washing step divided by the amount of monosulfuron added on the SPE column.

[b] Experiment was performed with SPE cartridge 1. In the SPE, 3 mL of washing solution and 5 mL of elution solvent (0.5% trifluoroacetic acid in MeOH) with a flow rate of 0.5 mL min^{-1} were used. The analytical condition used for the recovery determination is in Section 2.8.

the imprinted sections (with carboxyl groups) and the non-imprinted sections (polymer chain) of the MIP contributed to the binding of the template on the MIP stationary phase.

As a solvent with high elution strength for monosulfuron (Table 3), MeOH was chosen as the elution solution. It was found from the further optimization experiments that a shorter elution time and less tailing peaks for monosulfuron were obtained when the acidified MeOH was used as the eluting solution compared with MeOH. Because using trace amount of trifluoroacetic acid (TFA) is favorable for the evaporation after the SPE process, MeOH with 0.5% TFA was selected as the elution solution.

The eluting strength of different solvents for the soil impurities was also tested using the same conditions in Table 3. The background solution containing the soil impurities was loaded on the MISPE cartridge. The amount of impurities removed by the different solvent and solution was measured by HPLC and compared with the amount in the initial sample. Because the major components in the soil impurities are humic acids, which have similar polarities to monosulfuron, the order of eluting strength of tested washing solutions for the impurities was similar to that for the monosulfuron on the MISPE column. Although acetonitrile and acetone eluted less templates from the MIPSPE column, they also have weaker eluting ability for the soil impurities. Because in an aqueous buffer (pH 3.0)–MeOH mobile phase monosulfuron can be separated from soil impurities (Fig. 4) by the MIP stationary phase, aqueous buffer (pH 3.0)–MeOH was selected for the washing solution. The MISPE process using SPE cartridge 2 (Fig. 1) in the HPLC system was employed in the further MISPE condition optimization.

In the MISPE using SPE cartridge 2, water adjusted with H_3PO_4 (pH 3) was used to replace the pH 3.0 buffer in the washing solution to prevent the precipitation in the HPLC system when MeOH with 0.5% TFA (elution solution) was pumped in and mixed with washing solution. Washing step was optimized by selecting a water (pH 3.0)–MeOH ratio and washing volume. A solution of water (pH 3.0)–MeOH (7:3 (v/v)) was finally chosen for the washing step. The background solution spiked with monosulfuron was tested with the MISPE procedure to evaluate the sample clean-up efficiency of the process. The fraction (4 mL) from the elution step was collected and dried with an air stream. The residues was dissolved into 100 μL of water (pH 3.0)–MeCN (4:6 (v/v)) and analyzed with a C_{18} analytical column. After the SPE process, a clean baseline was obtained in the reversed-phase HPLC analysis (Fig. 6).

3.4. Accuracy and reproducibility of the SPE method

To determine the accuracy and precision of the MISPE method, background solutions with spiked monosulfuron of different concentrations were analyzed with the MISPE–HPLC procedure. The results of the experiment are shown in Table 4.

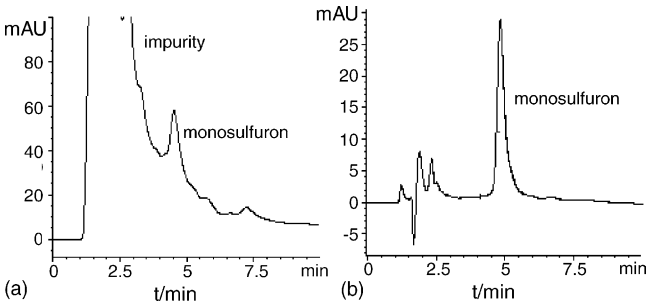

Fig. 6. The chromatograms of the extracts of monosulfuron spiked soil samples. In the HPLC analysis, a Zorbax C_{18} analytical column (150 mm × 4.6 mm) was used. The mobile phase was water (pH 3.0)–MeOH (55:45 (v/v)) with a flow rate of 1 mL min^{-1}. (a) The chromatogram of the original extract solution. (b) The chromatogram of the extract solution after the MISPE.

Table 4
Recoveries of monosulfuron in MISPE–HPLC analysis for the monosulfuron spiked soil extraction solution ($n = 3$)[a]

Concentration of spiked monosulfuron in background solution (μg mL^{-1})	Recovery, % (R.S.D., %)
5	97.1 (1.26)
10	95.1 (2.28)
20	95.5 (3.12)
25	95.3 (0.71)
30	96.8 (1.04)

[a] In the experiment, 10 g of soil sample was extracted and then spiked with monosulfuron for each measurement. The MISPE column (cartridge 2) was washed with water (pH 3.0)–MeOH (7:3 (v/v)) for 8 min, then back flashed with 0.5% TFA in MeOH to elute the monosulfuron. The analytical HPLC condition for recovery determination is in Section 2.8.

The data in Table 4 indicates that the MISPE method has good recoveries in the monosulfuron concentration range of 5 to 30 μg mL^{-1}. This MISPE method was selected for the sample clean-up in the monosulfuron residue analysis from the soil samples.

3.5. Analysis of soil samples

The reliability of the MISPE–HPLC method was evaluated with monosulfuron spiked soil samples. The monosulfuron was spiked into soil samples at different concentrations.

Table 5
Accuracy and precision of SPE–HPLC analysis for the monosulfuron spiked soil samples ($n = 5$)[a]

Concentration of monosulfuron spiked in soil (μg g^{-1})	Recovery, % (R.S.D., %)
2.5	93.5 (1.75)
1.5	96.7 (1.37)
1.0	96.2 (2.34)
0.25	109.9 (3.17)
0.1	98.4 (0.89)

[a] In each measurement, 10 g of soil sample was used for the fortification. In the SPE, the MISPE column (cartridge 2) was washed with water (pH 3.0)–MeOH (7:3 (v/v)) for 8 min, then back flashed with 0.5% TFA in MeOH to elute the monosulfuron. The analytical HPLC condition for recovery determination is in Section 2.8.

For each concentration five replicates of soil samples were prepared. After the solvent was evaporated, the soil samples were extracted and analyzed with the MISPE–HPLC procedure. The recoveries are listed in Table 5.

The average recoveries were above 93% for the samples with different monosulfuron-spike concentrations and the R.S.D. values were less than 3.2% (Table 5). The detection limit was $0.08\,\mu g\,g^{-1}$, when defined as three times of the noise of HPLC profile. The results demonstrated that the MISPE–HPLC method has good accuracy and precision for the determination of monosulfuron in soil.

4. Conclusions

Molecularly imprinted polymer with special selectivity was prepared for the soil sample clean-up in the monosulfuron residue analysis. The washing and elution solutions were selected based on the study of the influence of the mobile phase on the retention of the template and soil impurities on the MISPE column. With an efficient sample clean-up procedure, a practical method with good recovery and precision for the determination of monosulfuron in soil samples has been developed.

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