Headspace In-Tube Microextraction Combined with Reverse-Flow Micellar Electrokinetic Capillary Chromatography for Detection of Pyrethroid Herbicides in Fruits

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A rapid, simple, and sensitive method has been developed for the analysis of pyrethroid herbicides in fruits by using headspace in-tube microextraction (HS-ITME) coupled with reverse-flow micellar electrokinetic capillary chromatography (RF-MECC). In the newly developed method, by placing a capillary filled with background electrolyte (BGE) of RF-MECC in the HS above the sample solution, the pyrethroid herbicides were extracted into the acceptor phase in the capillary. After extraction, electrophoresis of the extracts in the capillary was carried out. The influence of some essential BGE components such as sodium dodecyl sulfate (SDS) and organic modifiers concentrations was investigated. Extraction parameters were also systematically investigated, including the extraction temperature, extraction time, salt concentration, and volume of the sample solution. Under the optimized conditions, enrichment factors for three pyrethroids were 309, 133, and 288, respectively. The proposed method provided a good linearity, low limits of detection (below 1.00 ng/mL), and good repeatability of the extractions (relative standard deviations [RSDs] below 7.83%, $n = 6$). The fruit samples were analyzed by the proposed method, and the obtained results indicated that the proposed method provides acceptable recoveries and precisions.

Keywords: Headspace in-tube microextraction, micellar electrokinetic capillary chromatography, pyrethroids, liquid microextraction

Introduction

Pyrethroids are man-made pesticides, with structures derived from naturally occurred pyrethroid found within *Chrysanthemum cinerariaefolium* flowers [1]. They are more frequently used in the cultivation of fruits and vegetables because of their low cost, efficacy at low dosage, and high environmental degradation rates when compared to other classes of pesticide [2, 3]. The widespread utilization and bioaccumulation of these compounds can present hazards to humans, and many organizations have set strict maximum residue limits for pyrethroid pesticides [4]. After high exposure to pyrethroid, humans showed mainly reversible and somehow unspecific symptoms like cough or respiratory irritation, dizziness, nausea, headache, irritation, vomiting, or paresthesia [5]. Nowadays, public interest in pesticide residues in foods and related commodities has been increased. This situation has led to regulation setting maximum residue limits (MRLs) of pesticide residues in agricultural products. Therefore, it is necessary to develop an analytical method with high sensitivity to meet the requirement for determination of pyrethroid in agricultural and food production.

Numerous methods have been reported for the determination of pyrethroid residues in agricultural commodities. Almost all of the analytical methods for pyrethroid residues are based on separation techniques such as gas chromatography (GC) [6, 7], high-performance liquid chromatography (HPLC) [8, 9], and capillary electrochromatography [10]. Sample pretreatment is a key step in the whole analysis process, especially for trace pyrethroid analysis. Till now, various sample preparation methods have been used for pyrethroid extraction, such as liquid-liquid extraction (LLE)

[11–13], solid-phase extraction (SPE) [14, 15], dispersive liquid-liquid microextraction (DLLME) [16, 17], quick, easy, cheap, effective, rugged and safe (QuEChERS) method [18], matrix solid-phase dispersion (MSPD) [19], suspended droplet microextraction (SDME) [20], hollow fiber liquid phase microextraction (HF-LPME) [21], headspace solid-phase microextraction (HS-SPME) [22], molecularly imprinted polymer silica monolith (MIPSM) [23], and so on. The advantages of the above techniques involve great efficiency, high analysis speed, and highly selective analyses. However, those methods require large amounts of organic reagents and tedious operation steps. The tedious operation steps are time-consuming. Furthermore, some of the methods require special procedures, such as centrifugation, vortexing, and ultrasonication; their utility is thus limited to laboratory conditions. Thus, simple sample preconcentration and rapid analysis technique is necessary for shorting the time for pyrethroid analysis.

Recently, owing to its high resolving power, low solvent consumption, and simple sample pretreatment, capillary electrophoresis (CE) has been used as an attractive method for herbicides residue analysis. However, due to its relatively low sensitivity, CE is not the most representative method among analytical separation techniques. Hence, to improve the CE sensitivity, various off-line or on-line methods have been developed prior to CE separation [24–27]. Recently, Lee et al. [28] reported a novel in-tube microextraction (ITME) combined with headspace (HS) extraction using the liquid inside the capillary as an acceptor phase, without forming a drop at the capillary tip. Then, the headspace in-tube microextraction (HS-ITME) in-line coupled with CE has been presented for an analysis of chlorophenols in an aqueous sample.

On the basis of previous research [29], in the present study, HS-ITME coupled with reverse-flow micellar electrokinetic

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capillary chromatography (RF-MECC) was for the first time applied for the extraction and determination of trace levels of three pyrethroids (cyfluthrin, fenpropathrin, tetramethrin) in fruit samples. In the proposed procedure, simply by placing a capillary filled with a running buffer in the HS above the sample solution, three analytes were extracted into the acceptor phase in the capillary. After extraction, electrophoresis of the extracts in the capillary was carried out. The obtained results indicated that the developed method is an excellent alternative for the routine analysis in the determination of pyrethroid.

Method

Reagent and Materials. The three pyrethroids of cyfluthrin, fenpropathrin, and tetramethrin (see Figure 1 for their chemical structures) were obtained from Aladdin Reagent Co., Ltd. (Shanghai, China). H_3PO_4 , NaOH, sodium dodecyl sulfate, acetonitrile, and 2-propanol were of analytical-reagent grade from Beijing Chemical Factory (Beijing, China). Deionized water was used throughout the experiment. The fruit samples were purchased from a common store (Qingdao of China). All solutions and samples were filtered through 0.45 μm syringe filter.

Apparatus and Chromatographic Conditions. A CL1030 capillary electrophoresis system (Beijing Lucai Scientific Instrument Co., Ltd., China) was employed throughout the experiment. The applied voltage was held constant at 30 kV. The column was an uncoated 50 μm ID fused-silica capillary of 85 cm and an effective length of 45 cm (Nuoxin, Hebei Province, China). The temperature of the capillary cartridge during electrophoresis was maintained at 25 $^\circ\text{C}$. Ultraviolet (UV) detection was set at 200 nm. A laboratory-made cover unit of a 10-mL sample vial consisted of a silicone cover and a section of 2-cm needle tube which was intercepted from 5-mL syringe.

Before use, the capillary was rinsed with 1 M NaOH for 10 min, with deionized water for 10 min and with methyl alcohol for 10 min; it was then conditioned with running buffer for 10 min. Between each run, the capillary was rinsed with electrolyte for 5 min. The optimal background electrolyte was composed of 120 mM sodium dodecyl sulfate (SDS), 50 mM H_3PO_4 , 15% acetonitrile, and 15% 2-propanol, at pH of 2.00.

Preparation of Standard Solutions and Fruit Samples. A standard solution of 1000 $\mu\text{g/mL}$ of each pyrethrin was prepared in acetonitrile. They were stored at 4 $^\circ\text{C}$, and the different concentrations of the sample solutions were prepared by appropriate dilution from the stock solution. About 5.0 g of sample flesh of fruit samples were homogenized and were extracted with 10 mL purified water for 1 h in an ultrasonic bath. For the matrix-matching between the standard series solution and real fruit samples, 5.1 M of NaCl was added to the fruit sample extract. The spiked samples at concentration levels of 5.0, 50, and 100 ng/mL for each individual pyrethrin were prepared by simultaneously spiking the standard solutions of the pyrethrin into the sample solutions and left alone for about 30 min. Then, the spiked samples were used for HS-ITME-CE analysis directly.

HS-ITME-RF-MECC Procedure. Figure 2 shows the device schematic of HS-ITME. Firstly, a 5.0 mL sample solution was transferred into the sample vial and 30% (w/v) sodium chloride was added into the sample solution while the capillary was washed. After washing steps, the separation capillary was filled with a running buffer, which could also be used as an acceptor phase. The inlet part of the separation capillary was placed in the center of the cover unit with the two slots at the same direction, and then the silicone plug was turned 180 $^\circ$ to seal the capillary in the cover unit. The sample vial was closed with the cover unit and the capillary tip was fixed at 1 cm height above the surface of the sample solution. Then, the extraction

Figure 1. Molecular structures of cyfluthrin, fenpropathrin, and tetramethrin

Figure 2. Device schematic of HS-ITME

was carried out at 90 °C for 15 min in the water bath on the magnetic stirrer at 600 rpm. After the extraction, the outlet and the inlet together with the cover unit and needle tube were placed in vials of running buffer put in positive and negative electrodes, respectively. Electrophoresis was then carried out.

Calibration of Sensitivity Enhancement Factor. The sensitivity enhancement factor (SEF) in terms of peak area can be calculated as the following equation: $SEF = A_{stack}/A$, where the A_{stack} is the peak area obtained with HS-ITME-RF-MECC and the A is the peak area obtained with the usual CE.

Results and Discussion

Optimization of the Separation Conditions. This experiment was done according to the author's previous study [30]. The

optimum conditions were an electrolyte containing 120 mM SDS, 50 mM H_3PO_4 , 15% acetonitrile, and 15% 2-propanol, at pH of 2.00. The applied voltage was held constant at 30 kV. The temperature of the capillary cartridge during electrophoresis was maintained at 25 °C. UV detection was set at 200 nm. Under the optimized conditions, the baseline separation of three pyrethroids was achieved in a relatively short time.

Optimization of Acceptor Phase. There were no electrophoresis behaviors of the three pyrethroids because of unionization of them in the solution. Thus, the anionic surface-active agent of SDS was added in the buffer to form micelle encapsulating the extracts so that the electrophoresis could be normally carried out. Moreover, organic modifiers were also employed according to hydrophobicity of the three pyrethroids

Figure 3. Effects of different factors on the extraction efficiency. Factors: (A) extraction time, (B) extraction temperature, (C) volume of sample, and (D) concentration of salt. 1, Cyfluthrin; 2, fenpropathrin; 3, tetramethrin. Analytical conditions: 120 mM SDS, 50 mM H_3PO_4 , 15% acetonitrile, and 15% 2-propanol, at pH of 2.00

so that the solubility of samples in the buffer was improved. A pH of 2.00 was selected to suppress electroosmotic flow (EOF). Therefore, the running buffer was used as the acceptor phase in this work. The evaporated analytes in the HS were extracted into the acceptor phase inside the capillary. The present scheme was extraordinarily simple and fully automatic.

Optimization of HS-ITME Procedure

Effect of Extraction Time. The extraction time has important effect on the extraction efficiency. In order to evaluate the effect of extraction time, extraction was carried out for 5.0, 10.0, 15.0, and 20.0 min, respectively. As shown in Figure 3A, the extraction efficiency increased with the extraction time before 15.0 min and then did not change with further increasing of the extraction time. Thus, the extraction time selected was 15.0 min.

Effect of Extraction Temperature. The evaluations of normal boiling points of three pyrethroids using ACD software (experimental measuring is impossible) give the values 496, 448, and 455 °C for cyfluthrin, fenpropathrin, and tetramethrin, respectively. Thus, the extraction temperature has great influence on the extraction efficiency of the pyrethroids. The effect of extraction temperature on the extraction efficiency was shown in Figure 3B. The result indicated that the extraction efficiency increased with the extraction temperature from 50 °C to 90 °C. The partitioning of an analyte between the donor phase and the HS at equilibrium is represented by Henry's constant in relation to the partial pressure of the analyte vapor in the HS. For a volatile analyte, the evaporation from the donor phase to the HS is an endothermic process. Thus, as the temperature is increased, Henry's constant will become larger, that is, more of the analyte will evaporate into the HS. However, the electric current interrupted and separation could not be carried out at 100 °C. Thus, we selected 90 °C as the optimal temperature.

Effect of Sample Volume. The volume of sample solutions can influence the equilibrium concentration of analytes in acceptor phase and the enrichment factor of analytes in HS-ITME. The effect of sample volume was investigated in the range from 2.0 to 6.0 mL, and the result was shown in Figure 3C. Experiments demonstrated that for the three pyrethroids the extraction efficiency increased with the sample volume up to 5.0 mL and followed by decreasing with further increasing of the sample volume. When sample volume was less than 5.0 mL, with increasing of sample volume, the solutes in sample solution become more and tend to diffused into extraction phase. Whereas, with increasing sample volume further, the analyte vapor into headspace was limited so as to extraction efficiency decrease. Therefore, 5.0 mL was adopted as the final sample volume in the subsequent studies.

Effect of Salt Concentration. Salt-out effect is widely used in LPME to increase the extraction efficiency. In this study, the effect of the ionic strength of sample solution on extraction efficiency was investigated by using different concentration of NaCl (1.7, 3.4, 5.1, 6.2 M). As shown in Figure 3D, the peak area of the three pyrethroids achieved a maximal value with 5.1 M NaCl, resulting from the interaction of water molecules with analytes and salt ions. Hydration spheres around the salt ions formed by water molecules could reduce the water concentration to dissolve the pyrethroid and be beneficial to remove the pyrethroid into the headspace. However, the interactions between salt ions and analyte molecules were also accelerated with the NaCl concentration higher than 5.1 M, which reduced the pyrethroid evaporation into the headspace.

From the above results, the optimized HS-ITME conditions were as follows: extraction time, 15 min; extraction temperature, 90 °C; sample volume, 5.0 mL; and salt concentration, 5.1 M. Under the optimum HS-ITME–RF-MECC conditions, the typical electropherograms of standard mixture under the normal and

stacking conditions are shown in Figure 4 (d and e), and the stacking enhancement factors of the three pyrethroids are listed in Table 1.

Evaluation of Method Performance. To investigate the method performance of the proposed method for determining pyrethroid, a series of experiments was designed for evaluating the parameters including linearity, reproducibility, limits of detection, and other characteristics of method under the optimized condition. Table 1 shows them. Linearity was observed in the range of 2.0–100 ng/mL for cyfluthrin, 0.05–50 ng/mL for fenpropathrin, and 5.0–100 ng/mL for tetramethrin, respectively. Although the solubility of cyfluthrin and fenpropathrin is 2 ng/mL and 14 ng/mL, respectively, we believe that the pyrethroid solubility in water can be increased by such conditions as acetonitrile, heating, and agitation. In the published analytical methods, the maximum concentration of pyrethroid in water was 400, 500, 500, and 100 ng/mL, respectively [14–17]. All the analytes exhibited good linearity with correlation coefficient (r) ranging from 0.9908 to 0.9985. The proposed method provided a good repeatability of the extractions (relative standard deviations [RSDs] below 7.83%, $n = 6$). The relative RSDs of the migration time and the peak area of the analytes were lower than 4.75% and 5.63% for intra-day and inter-day. The detection limits ($S/N = 3$) of cyfluthrin, fenpropathrin, and tetramethrin were 0.5, 0.01, and 1 ng/mL, respectively. The limit of quantification ($S/N = 10$) of cyfluthrin, fenpropathrin, and tetramethrin was 2.0, 0.04, and 3.0 ng/mL, respectively. The recoveries of the target compounds were determined in the fruit samples spiked at three concentration levels (5.0, 50, and 100 ng/mL), and good spike recoveries (92.31–103.75%, RSDs below 6.93%, $n = 6$) were achieved for all three pyrethroids. These parameters indicated that the present approach with high sensitivity and reliability could be used to detect the concentration of pyrethroid.

Figure 4. The typical electropherograms of standard mixture of pyrethroids and real samples. 1, Cyfluthrin; 2, fenpropathrin; 3, tetramethrin. a, Blank peach sample; b, peach sample; c, crab apple sample; d, standard mixture of pyrethroids under normal condition; e, standard mixture of pyrethroids under HS-ITME-CE. Analytical conditions: 120 mM SDS, 50 mM H_3PO_4 , 15% acetonitrile, and 15% 2-propanol, at pH of 2.00, applied voltage of 30 kV, capillary temperature of 25 °C, and UV detection of 200 nm. (a, b, c, e) Extraction conditions: the running buffer of RF-MECC as for acceptor phase; extraction time, 15 min; extraction temperature, 90 °C; sample volume, 5.0 mL; salt concentration, 5.1 M; and stirring rate at moderate speed. The final concentrations of analytes were 2.0, 0.5, and 5.0 ng/mL for cyfluthrin, fenpropathrin, and tetramethrin, respectively. (d) Analytical conditions such as (a); the standards solution were diluted with methyl cyanide and then injected for 10 s with the height difference of 20 cm between the inlet end and the outlet end of capillary. The final concentrations of analytes were 20, 2.5, and 50 ng/mL for cyfluthrin, fenpropathrin, and tetramethrin, respectively.

Table 1. The performance characteristics of the proposed method

Analytes	Linear range (ng/mL)	Regression equation	<i>r</i>	LOD (ng/mL)	LOQ (ng/mL)	Recoveries (%)	RSD (%)	SEF
Cyfluthrin	2.00–100	$y = 120.33x + 241.4$	0.9984	0.50	2.00	92.31	4.75	309
Fenpropathrin	0.05–50	$y = 608.74x + 661.69$	0.9908	0.01	0.04	102.45	5.58	133
Tetramethrin	5.00–100	$y = 6.7731x + 720.54$	0.9985	1.00	3.00	103.75	5.63	288

Table 2. Comparison of the developed method with other reported methods for the determination of pyrethroids

Method	Linearity range (ng/mL)	LOD (ng/mL)	LOQ (ng/mL)	Recoveries (%)	SEF	Ref.
On-site DLLME-SSS-HPLC-UV	1–500	0.24–0.68	–	84.7–95.3	121–136	[16]
Hydrazone COF-SPME-GC-ECD	1–1000	0.11–0.20	0.37–0.77	75.6–106.4	307–2327	[22]
SPLE-GC-MS	1–400	1.00–5.00	–	94.3–103	–	[14]
DLLME-SFO-GC-ECD	5–100	0.08–0.5	–	92.1–99.6	292–888	[17]
SPE-HPLC-UV	30–500	0.05–10	–	69–131	98–1025	[15]
HS-ITME-CE	0.05–100	0.01–1.00	0.04–3.00	92.31–103.75	133–309	This work

Application to Fruit Samples Analysis. The developed HS-ITME-CE method was applied to determine the three pyrethroids in fruit samples were analyzed. There was no target pyrethroid that could be detected in fruit samples. In order to illustrate the effect of matrix, the target compounds were determined in the fruit samples spiked at 2.0, 0.5, and 5.0 ng/mL for cyfluthrin, fenpropathrin, and tetramethrin. The typical chromatograms of blank and spiked peach sample are shown in Figure 4 (a and b). The typical chromatograms of blank and crab apple sample are shown in Figure 4 (a and c). The results revealed the feasibility of the HS-ITME-CE method.

Comparison of HS-ITME-CE with Other Analytical Methodologies. A comparison of the proposed method with the published analytical methods for the determination of the target pyrethroid is summarized in Table 2. The data shows that the proposed method possesses several advantages. Due to target pyrethroid obtained by in-tube microextraction, less organic solvent is required. In addition, the centrifugation process was avoided and reduced experiment time. Similar or better results were found in terms of the linearity, LOD, and recovery. These results demonstrate that the HS-ITME-CE method is simple, effective, inexpensive, and environmentally friendly.

Conclusions

In this study, a novel HS-ITME-CE method was developed for the determination of three pyrethroids in fruit samples. ITME uses the liquid in the separation capillary as an acceptor phase and thus utilizes all of the extracts for the subsequent CE analysis. The advantages of the new HS-ITME-CE lie in excellent stability compared with the conventional SDME-CE. It offers a quite powerful but extremely easy method readily usable by anyone without special equipment or training. After the optimization of the extraction and separation conditions for HS-ITME-CE, an effective enrichment procedure with good linearity and satisfactory recoveries was obtained. The obtained results indicated that the developed method is an excellent alternative for the routine analysis of pyrethroid in the food field.

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Conflict of Interest

The authors declare that there is no conflict of interest associated with this work.

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