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Simultaneous determination of cypermethrin and permethrin in pear juice by ultrasound-assisted dispersive liquid-liquid microextraction combined with gas chromatography

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ABSTRACT

A new method was developed for simultaneous determination of cypermethrin and permethrin residues in pear juice with ultrasound-assisted dispersive liquid-liquid microextraction (UA-DLLME) and gas chromatography-flame ionization detection (GC-FID). 3.5 mL of methanol (dispersant) and 30 μL of C_2Cl_4 (extractant) were injected into 5.0 mL of pear juice sample and then emulsified by ultrasound for 2.0 min to forming the cloudy solution. Under the optimum condition, the enrichment factors for cypermethrin and permethrin were 344 and 351 fold respectively. Good linearity was observed in a range of 0.009-1.52 μg g^{-1} with the correlation coefficient (r^2)≥0.9993. The limits of detection (LODs) were 3.1 and 2.2 μg kg^{-1} for cypermethrin and permethrin, respectively (S/N = 3). The recoveries of the method evaluated at three spiked levels were in the range of 92.1%-107.1%. The repeatability evaluated as intra-day and inter-day precision (RSDs) were less than 4.0% (n = 5). The developed method was successfully applied to determine the two pesticide residues in different pear juice samples.

1. Introduction

Cypermethrin and permethrin are two kinds of pyrethroid pesticides, which are widely used to prevent and treat insects both in daily life and in agriculture due to their broad spectrum insecticidal capacity and high effectiveness [1,2]. The wide applications of these pesticides provide benefits for increasing agricultural production, but by bioaccumulation and residual toxicity through the food chain, they can eventually become a risk or threat to both animal and human life [3–5]. Their residues in fruits, vegetables and other agricultural products will represent a serious hazard to human health such as cancer, infertility, nerve disorders, immunological and respiratory diseases [6]. Thus, there is a growing interest in the development of fast and reliable analytical procedure for extraction and trace-level determination of these pesticides in agriculture and food production. Many countries have made severe restrictions on their residues in fruit and vegetable products, for example, the Administration of China stipulates the residue limits for cypermethrin and permethrin in pear and other common fruits were less than 2.0 mg kg^{-1}.

Owing to the complexity of sample matrices and the relative low concentration of the target analytes in fruits and vegetables, sample pretreatment process is a crucial step in the analytical procedure to obtain accurate and sensitive results. Even with the advent of advanced hyphenated techniques based on mass spectrometry, the complex real matrices usually require extensive extraction and purification [7]. Currently, the pretreatment methods mainly involve the use of one or the combination of the following techniques for both the sample extraction and clean-up steps: liquid-liquid partitioning [8], solid-phase extraction [9,10], gel-permeation chromatography [11], matrix solid-phase dispersion extraction [12], solid-phase microextraction [13], liquid-phase microextraction [14], emulsification-mixroextraction [15,16] etc. However, from the practical point of view, these pretreatment procedures suffered from several inherent defects, such as non-equilibrium extraction procedures or the small contact surface between the phases, which negatively affects the sample throughput and the enrichment factors.

Recently, Rezaee et al. [17] developed a novel microextraction technique, termed dispersive liquid-liquid microextraction (DLLME), which is based on a ternary component solvent system like homogeneous liquid-liquid extraction and cloud point extraction. In this method, the appropriate mixture of extraction solvent and dispersive solvent are injected rapidly into an aqueous sample by syringe, resulting in the formation of a cloudy solution, which can markedly increase the contact surface between phases and reduce the extraction time with high enrichment factors [18]. DLLME technology combines extraction and concen-

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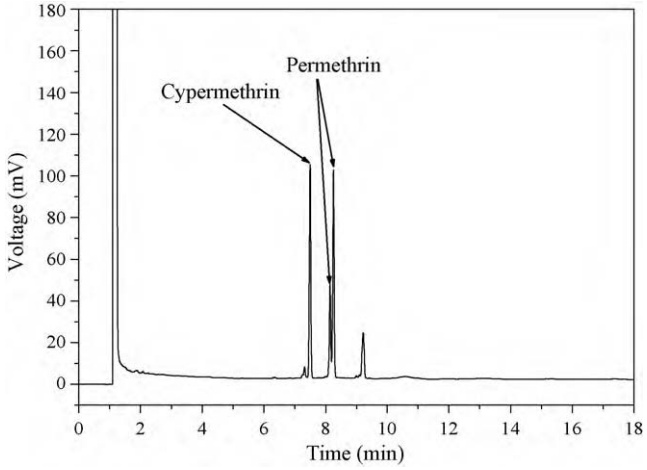

Fig. 1. Chromatogram of cypermethrin and permethrin solution.

tration in one step, and it has been used for the determination of different pollutants such as polycyclic aromatic hydrocarbons [19], organophosphorous pesticides [20], chlorobenzenes [21], chlorophenols [22], phenols [23], triazine herbicides [24], amide herbicides [25], pyrethroid pesticides [26] and metal ions [27–29] in water samples. However, its application for other samples more complicated than water, such as wine [30], honey [31], milk [32], fruits [33] and vegetables [34] is still in the exploratory stage. Moreover, these applications still encountered some drawbacks, such as low repeatability and enrichment factors, complicated pretreatment and clean-up steps, etc.

The aim of this study was to develop a simple and rapid UA-DLLME method for the extraction and determination of cypermethrin and permethrin in pear juice by using methanol as pretreatment reagent and dispersant. An ultrasound-assisted process was applied to accelerate the formation of the fine cloudy solution, which obviously increased the extraction efficiency and reduced the equilibrium time. Various parameters affecting the extraction and enrichment efficiency were evaluated and optimized. Under optimum condition, the enrichment factors for cypermethrin and permethrin were 344 and 351 fold, respectively.

2. Experimental

2.1. Reagents and standards

Chlorobenzene (C_6H_5Cl), 1,2-dichloroethane ($C_2H_4Cl_2$), tetrachloroethylene (C_2Cl_4), chloroform ($CHCl_3$), dodecanol, tetrachloroethane ($C_2H_2Cl_4$), and tetrachloromethane (CCl_4) were all analytical grades and purchased from Huaxin Chemical Reagent Co. (Baoding, China). Methanol, ethanol, acetone, tetrahydrofuran (THF), acetonitrile, isopropanol, acetic acid and sodium hydroxide were all analytical grades and purchased from Kermel Chemical Co. Ltd. (Tianjin, China). Cypermethrin and permethrin were obtained from Yangnong Chemical Co. Ltd. (Yangzhou, China). All the other reagents used in the experiment were of the highest grade commercially available. Double deionized water was filtered with 0.45 μm filter membrane before use.

2.2. Instrumentation and conditions

The chromatographic analysis was carried out on a Shimadzu GC-2014 system equipped with a split/splitless injector and an FID detector (Shimadzu, Japan). High-purity nitrogen (99.999%) was used as carrier gas and a GH-300 high-purity hydrogen generator and GA-2000A air pump (Beijing ZXHL Technology Development Co. Ltd.) were used to supply hydrogen and oxygen at the rate of 40 mL min^{-1} and 400 mL min^{-1}, respectively. The capillary column was KB-1 (100% dimethylpolysiloxane, 30 m × 0.25 mm × 0.25 μm) and its column flow rate was set at 1.5 mL min^{-1} with a split ratio of 10:1. An N-2000 data workstation (Zheda Zhineng Co. Ltd., Hangzhou, China) was used as the data acquisition system. The temperature-programmed mode was as follows: the initial oven temperature was set at 230 °C for 5 min, and then ascended to 285 °C at the rate of 20 °C min^{-1} and held for 10 min. The injection port and detector temperatures were maintained at 290 °C and 300 °C, respectively. The chromatogram of the standard solution was shown in Fig. 1.

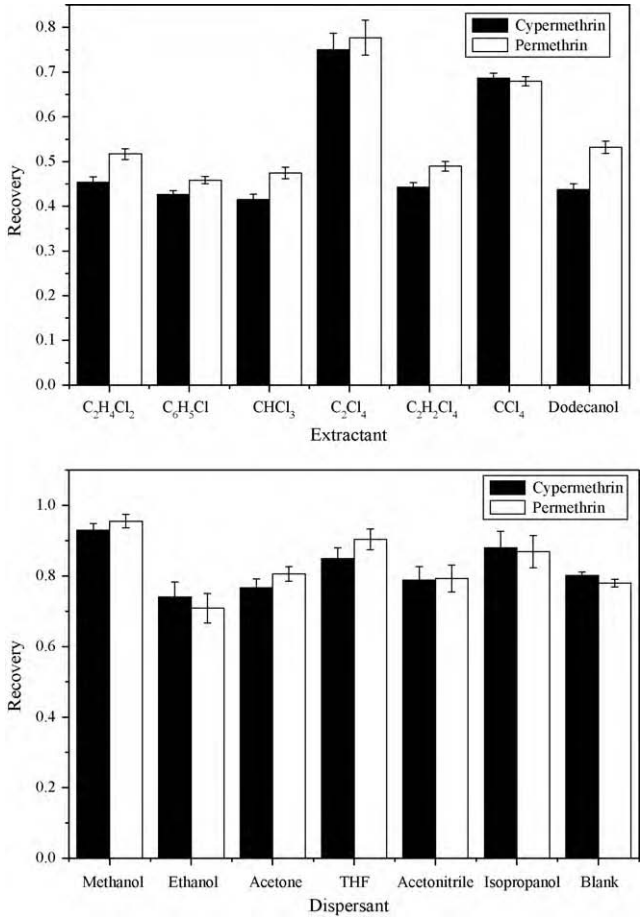

Fig. 2. The effect of extractant and dispersant on the ERs of UA-DLLME.

2.3. Sample preparation and UA-DLLME procedure

Pear samples purchased from different local markets (Baoding, China) were juiced by a food squeezer. The pear juice was filtrated under vacuum and subsequently centrifuged at 4000 rpm for 10 min. 5.0 mL of the supernatant liquid was transferred into a conical centrifuge tube and mixed with 3.5 mL of methanol. After shaking and filtrating of the sample solution, 30 μL of C_2Cl_4 was added to forming the ternary solvent system. The ternary mixture was vortex oscillated for 30 seconds and then further emulsified by ultrasound for 2.0 min to get the fine cloudy solution. Finally, the ternary solution was centrifuged at 4000 rpm for 5.0 min to get the sediment phase at the bottom of the centrifuge tube. The upper aqueous phase was removed with a syringe, and the sediment phase was used for further chromatographic analysis.

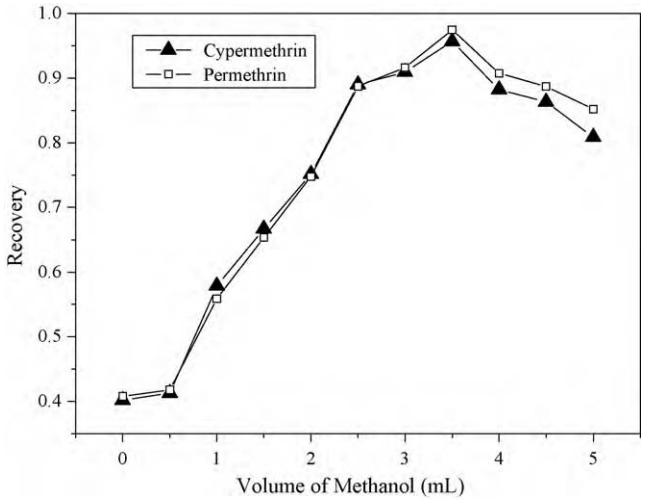

Fig. 3. The effect of methanol volume on UA-DLLME.

3. Results and discussion

3.1. Optimization of the UA-DLLME condition

In the UA-DLLME method, there are several factors that would significantly affect the extraction efficiency, such as the types and volumes of extractant and dispersant, ultrasonic time and pH of the solution. It is necessary to optimize these parameters so as to obtain the maximum extraction performance. In this work, the enrichment factor (EF) and extraction recovery (ER), respectively characterizing the performance of concentration and the extraction efficiency, were employed for the evaluation of the proposed UA-DLLME. The EF was defined as the ratio between the concentration of analyte in the sediment phase (C_{sed}) and the initial concentration of analyte (C_0) in the sample: $EF = C_{sed}/C_0$. The ER was defined as the percentage of the total analyte (n_0) that was extracted to the sediment phase (n_{sed}): $ER = n_{sed}/n_0 \times 100 = C_{sed} \times V_{sed}/C_0/V_{aq} \times 100$, where V_{sed} and V_{aq} were the volumes of sediment phase and sample solution, respectively. For the selection of extractant and dispersant, the standard solution was used directly as the donor phase, whereas the rest parameters were investigated on the pear juice samples spiked with the standard solution.

3.1.1. Selection of extractant

The type of extractant used in UA-DLLME is the essential consideration for efficient extraction. It should have higher density than water and high extraction capability for the target compounds and low solubility in water. Based on the above point of view, different kinds of extractants including chlorobenzene, 1,2-dichloroethane, tetrachloroethylene, chloroform, dodecanol, tetrachloroethane and tetrachloromethane were studied. Since these extractants had different solubility in aqueous phase, according to the relationships between the extractant volumes and the sediment phase volumes, which were investigated through adjustments and measurements by microsyringes, different volumes of each extractants with 0.5 mL of acetone were added into 5.0 mL of sample solution to achieve 11 μL of sediment phase, respectively. For dodecanol as extractant, it floated on the surface of the aqueous solution after centrifuging due to its lower density than water. The centrifuge tube was put in refrigerator for 5.0 min to curdle the dodecanol droplets (melting point: 24 °C), then the solid droplets were collected and melted for GC analysis. Fig. 2 indicated that C_2Cl_4 had the highest ERs among the seven extractants with a relatively small dosage. Therefore, C_2Cl_4 was selected as the extractant for this work.

3.1.2. Selection of dispersant

As the dispersant in UA-DLLME, it should be quite miscible in both the organic phase (extractant) and the aqueous phase (juice sample), so that it can disperse the droplets of extractant into the sample phase and increase the surface area between the phases for the mass transferring of target compounds, accordingly improve the extraction efficiency. Thus, methanol, ethanol, acetone, THF, acetonitrile and isopropanol as disperser solvents were investigated and compared. As had been reported by Liang et al. [35], different kinds of dispersants resulted in volume change of the sediment phase even with the constant volume of extractant, which were due to its different solubility in extractant and aqueous solution. Therefore, 0.5 mL of each dispersant (methanol, ethanol, acetone, THF, acetonitrile and isopropanol) with different volumes of C_2Cl_4 were applied for 5.0 mL sample solution to achieve a constant volume of sediment phase (11 μL), respectively. According to the results in Fig. 2, the highest ERs were obtained using methanol as dispersant, which may be due to its higher dispersing capability for the extractant and relatively less loss for the analytes.

3.1.3. Effect of methanol volume

The volume of dispersant is one of the key parameters of the UA-DLLME procedure. It related to extraction efficiency and the formation of cloudy solution, especially in the present work, where the dispersant was also used as the pretreatment reagent to prevent the endogenous interferences precipitate. In order to study the effect of methanol volume on the extraction performance, different volumes of methanol in a range of 0-5.0 mL were investigated. The results showed that the volume of sediment phase increased with the increase of methanol from 0 to 2.5 mL and then decreased when the volume of methanol further increased from 2.5 to 5.0 mL. It was due to the two competitive effects: one was methanol being miscible with C_2Cl_4 to increase the volume of sediment phase, and another was it enhanced the solubility of extractant in aqueous phase so as to reduce the volume of sediment phase. At the same time, the EFs increased with the increase of methanol volume, which was because methanol could disperse C_2Cl_4 into sample solution to get more fine droplets, therefore, increase the contact surface and gain higher extraction efficiency. Furthermore, the reduced volume of sediment phase with the increasing volume of methanol also led to the increase of the EFs. In Fig. 3, a gradual increase of ER was observed with increase of methanol volume from 0 to 3.5 mL, which was owing to the contribution of methanol in eliminating the interferences of sample matrixes as well as in dispersing the extractant. However, further increase of methanol volume beyond

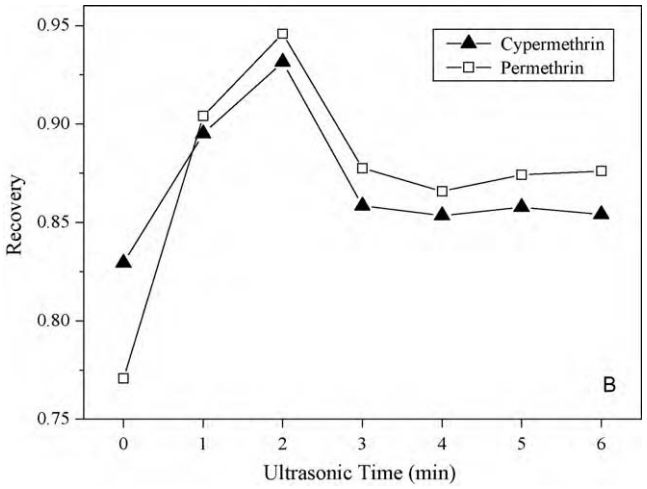

Fig. 4. The effect of ultrasonic time on the ERs of UA-DLLME.

Table 1
Parameters of the UA-DLLME-GC method.

Compounds	RSD[a] (%)	RSD[b] (%)	EF[c]	Linearity ($\mu g\ g^{-1}$)	Linear equation[d]	r^2	LOD ($\mu g\ kg^{-1}$)
Cypermethrin	3.8	3.4	344	0.009-1.52	$y = 5.14 \times 10^5 x - 140.53$	0.9993	3.1
Permethrin	4.0	3.4	351	0.009-1.52	$y = 7.47 \times 10^7 x - 41.09$	0.9995	2.2

[a] Intra-day relative standard deviation, n = 5.
[b] Inter-day relative standard deviation, n = 3.
[c] Average enrichment factor, n = 7.
[d] y: analyte peak area; x: analyte concentration ($\mu g\ g^{-1}$).

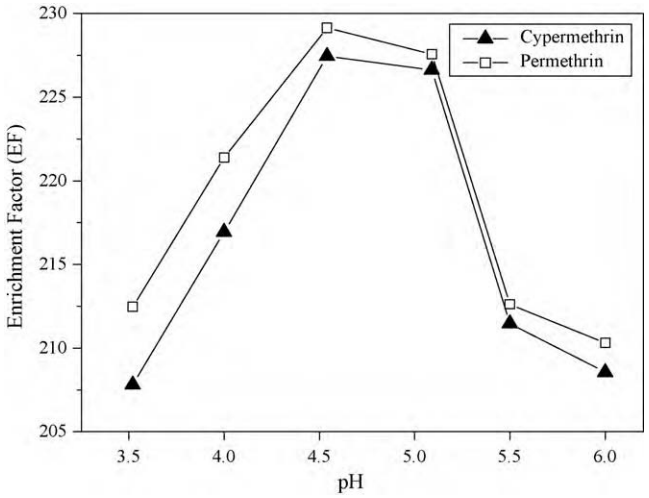

Fig. 5. The effect of sample pH on the EFs of UA-DLLME.

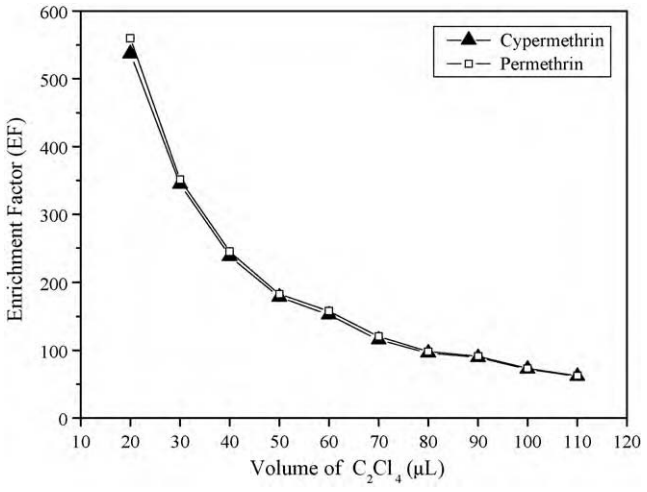

Fig. 6. The effect of C_2Cl_4 volume on the EFs of UA-DLLME.

3.5 mL caused a decreasing ER, which also resulted from the dramatic reduction of sediment phase volume (methanol enhanced the solubility of extractant in aqueous phase so as to reduce the volume of sediment phase). Therefore, 3.5 mL of methanol was selected for this work.

3.1.4. Effect of ultrasonic time

Ultrasound can accelerate the formation of fine cloudy solution, which would markedly increase extraction efficiency and reduce equilibrium time. Therefore, an ultrasound-assisted process was adopted in a range of 0-6.0 min to evaluate its effect on extraction performance. The results revealed that the extension of ultrasonic time within 2.0 min resulted in obvious increase of EFs and ERs, which was due to the assisting-dissolving and emulsifying effect of the ultrasound-assisted process. However, the ERs decreased when further prolong the ultrasonic time over 2.0 min (Fig. 4). The reason was due to the fine droplets of C_2Cl_4 were formed to increase the contact surface of the two phases within 2.0 min and therefore accelerated the analytes transferring into the extractant; however, the volatilization loss of the anaytes and extactant under ultrasound increased with the extension of ultrasonic time, which resulted in a reduced EF and ER in the range of 2.0-6.0 min. Therefore, 2.0 min was selected as the optimum ultrasonic time.

3.1.5. Effects of salt concentration and the pH of sample

Considering the salting out effect (commonly sodium chloride) had been used in DLLME to improve the extraction of analytes from water samples, different amounts of sodium chloride in a range of 1%-30% (w/v) were investigated in UA-DLLME procedure. The results showed that much flocculation precipitate was observed at the bottom of the centrifuge tube, which was caused by the precipitation of pear juice matrixes under salting out effect. Thus, salting out effect was not applied for the UA-DLLME procedure.

The pear juice is weak acidity and containing much endogenous interference, which have negative impact for the UA-DLLME. To investigate the effect of pH of sample solution on extraction performance, the pH of juice samples was adjusted in a range of 3.5-7.0 using acetic acid and sodium hydroxide solution (Fig. 5). The highest EF and ER were obtained at pH 4.5 and no sediment phase was observed when the pH was over 6.5. Therefore, pH 4.5 was suitable for the UA-DLLME procedure.

3.1.6. Effect of extractant volume

In UA-DLLME, the volume of organic extractant was one of crucial parameters that had an important effect on the extraction efficiency. The volume of extractant was expected as less as possible to achieve the highest EF and the lowest toxicity for environment; on the other hand, it should be suitable to extract the analytes as much as possible and ensure enough sediment phases for further chromatographic analysis. Therefore, the volume of C_2Cl_4 in a range of 10-110 μL was investigated and the results revealed that the EFs continuously increased with the decrease of C_2Cl_4 volume in the test range (Fig. 6). However, the volume of sediment phase decreased with the reducing volume of C_2Cl_4. When the volume of C_2Cl_4 was less than 30 μL, the sediment phase was too small for quantitative analysis and the RSD increased obviously. On the other hand, the ERs increased rapidly to over 92% when the C_2Cl_4 volume

Table 2
Comparison of the present technique with other reported methods.

Parameter		Value/and remark	
		Reported method	Present method
Amount of organic solvent (mL)		240[a], 170[b], 145[c]	3.53
Concentration means		Evaporation[a,b,c]	No evaporation
LOD ($\mu g\ kg^{-1}$)	Cypermethrin	10[a], 20[b], 15[c]	3.1
	Permethrin	5[a], 30[b], 15[c]	2.2

[a] Reference [36].
[b] Reference [37].
[c] Reference [38].

Table 3
Recoveries of the UA-DLLME-GC method.

Compounds	sample concentration ($\mu g\,g^{-1}$)	Spiked concentration ($\mu g\,g^{-1}$)	Found concentration ($\mu g\,g^{-1}$)	Average recovery (%)	RSD (n = 3) (%)
Cypermethrin	ND[a]	0.014	0.015	107.1	1.6
		0.379	0.349	92.1	1.1
		0.947	0.912	96.3	3.7
Permethrin	0.016	0.014	0.031	107.1	3.4
		0.379	0.372	93.9	7.0
		0.947	0.912	94.6	3.1

[a] ND, not detected.

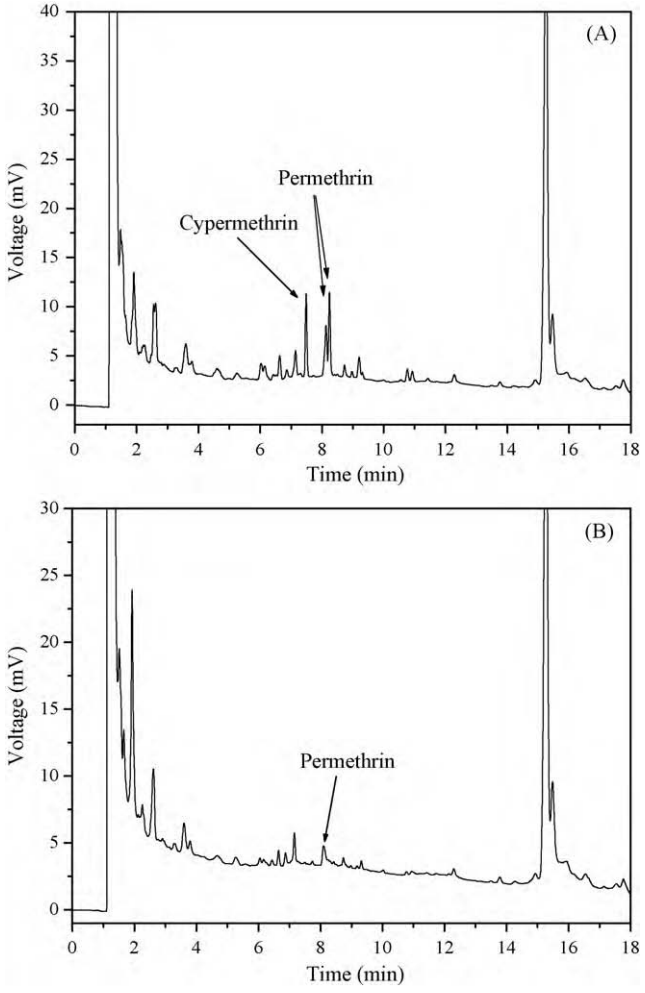

Fig. 7. Chromatograms of spiked (A) and real pear juice sample (B).

increasing to 30 μL and then only slightly increased (98%) even further increase the volume of C_2Cl_4 to 110 μL. Considering the EF, ER, sediment volume and reproducibility, 30 μL of C_2Cl_4 was used as extractant in the subsequent experiments.

3.2. Evaluation of the UA-DLLME-GC method

3.2.1. Features of the method

To evaluate the developed UA-DLLME-GC method, the linearity, precision, repeatability, enrichment factors and limits of detection were investigated under the optimum condition. Matrix-matched calibration curves were constructed using the areas of the chromatographic peaks measured at eight increasing concentrations, in a range of 0.009-1.52 $\mu g\,g^{-1}$. Good linearity was observed for the two analytes throughout the concentration range, and the regression equations were shown in Table 1. The precision and accuracy were determined by analyzing five replicates of the spiked samples (0.95 $\mu g\,g^{-1}$) on the same day and three different days. Intra-assay and inter-assay precisions expressed as the relative standard deviations (RSDs) were less than 4.0% and 3.4%. Under the optimum condition, the enrichment factors for cypermethrin and permethrin were 344 and 351 fold. The LODs calculated on the basis of signal-to-noise ratio of 3 were 3.1 and 2.2 $\mu g\,kg^{-1}$, respectively. The comparison of the present technique with other reported methods in terms of the amount of organic solvent, concentration means and LODs was provided in Table 2.

3.2.2. Application of the technique

The applicability of the UA-DLLME-GC method was verified by determination of cypermethrin and permethrin in different pear juice samples (Fig. 7). All the samples collected from local markets were extracted according to the section 2.3. Trace amount of permethrin was detected in five pear samples at levels of 3.9-16.4 $\mu g\,kg^{-1}$, which were below the maximum residue limits established by the Standardization Administration of China. To study the effect of sample matrix and the accuracy of the UA-DLLME method, recovery experiments were carried out by spiking three different concentrations of standard analytes (0.014, 0.379 and 0.947 $\mu g\,g^{-1}$) into pear juice samples (Table 3). The recoveries of cypermethrin and permethrin were in the range of 92.1%-107.1%, which indicated that the method was reliable and could be used for the trace analysis of the two pyrethroid residues in pear juice samples.

4. Conclusion

This work demonstrated the successful development of UA-DLLME-GC method for the extraction and determination of two representative pyrethroid residues in pear juice samples. An ultrasound-assisted process was applied to accelerate the formation of a fine cloudy solution, which markedly increased extraction efficiency and reduced equilibrium time. Under the optimum condition, the enrichment factors for cypermethrin and permethrin were 344 and 351 folds. The developed method has the advantages of high enrichment factor and sensitivity, low cost and easy operation.

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