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ISSN 1615-9306 · JSSCCJ 45 (14) 2022 · Vol. 45 · No. 14 · July 2022

Journal of SEPARATION SCIENCE

14|2022

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WILEY-VCH

Received: 21 February 2022 | Revised: 2 May 2022 | Accepted: 2 May 2022

DOI: 10.1002/jssc.202200147

RESEARCH ARTICLE

JOURNAL OF SEPARATION SCIENCE

Research method of rapid determination of chiral pesticide fenpropathrin enantiomers in fruit and vegetable puree by supercritical fluid chromatography

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Funding information
National Program on Key Research Project of China, Grant/Award Number: 2019YFE0103900

A method is first established for the separation and determination of fenpropathrin enantiomer residues in apple puree, strawberry puree, and tomato puree considered a supplementary food for infants by supercritical fluid chromatography. After the sample was extracted with acetonitrile and cleaned up by a solid-phase extraction column, then it was separated by a CHIRALPAK AD-3 chiral column with gradient elution at a flow rate of 1.5 mL/min using methanol and supercritical carbon dioxide as the mobile phase, detected by ultraviolet detector at 230 nm wavelength and quantified with the external standard method. The limits of quantification of the two fenpropathrin enantiomers were both 0.2 mg/kg, the linear ranges were 1.0–20.0 mg/L with linear correlation coefficients greater than 0.9992, the recoveries in the spiked samples at 0.2, 0.4 and 2.0 mg/kg were from 80.6 to 105%, and the relative standard deviation reached 2.6–7.7%. This method has the advantages of convenient operation, good resolution, and environmental protection, which can satisfy the requirement of determination for fenpropathrin enantiomer residues in fruit and vegetable puree as supplementary food for infants.

KEYWORDS
chiral separation, enantiomers, fenpropathrin, pyrethroid, supercritical fluid chromatography

1 | INTRODUCTION

Fenpropathrin, whose chemical name is α-cyano-3-phenoxybenzyl-2,2,3,3-tetramethylcyclopropaneate, is an important pyrethroid insecticide and acaricide with moderate toxicity and a wide insecticidal spectrum, which is widely used in the prevention and control of diseases of fruits and vegetables, such as citrus, apples, lychees and so on. Its extensive use on food and environmental pollution issues has also attracted widespread attention. Studies have shown that fenpropathrin has a chiral carbon atom, and a pair of enantiomers whose structure is (−)-fenpropathrin and (+)-fenpropathrin [1–3] (Figure 1).

Large differences in insecticidal activity and degradation rate between different fenpropathrin enantiomers, with (+)-fenpropathrin showing higher insecticidal activity than (−)-fenpropathrin and a faster degradation rate [1]. In agricultural production, the use of a single high-activity (+)-fenpropathrin will help reduce the use of fenpropathrin and reduce the pollution of the ecological

Abbreviation: HLB, hydrophile lipophile balance.

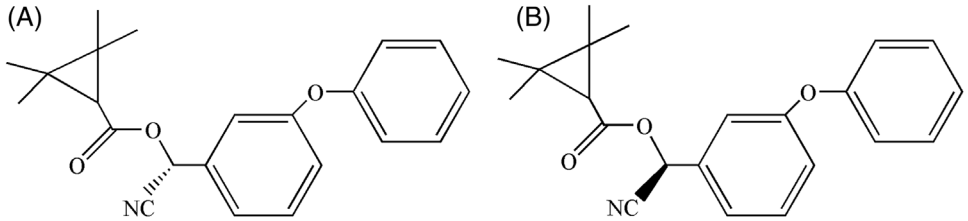

FIGURE 1 The chemical structures of different fenpropathrin enantiomers. (A) (−)-Fenpropathrin and (B) (+)-Fenpropathrin

TABLE 1 The maximum residue limits of fenpropathrin in apple, strawberry, and tomato matrixes

Matrix	Japan (mg/kg)	Korea (mg/kg)	China (mg/kg)
Apple	5	1	5
Strawberry	5	2	2
Tomato	2	2	1

environment by pesticides, which are still produced and used in the form of racemate at present.

Maximum residue limits for fenpropathrin in fruits and vegetables have been established in various countries for controlling the standardized use of such pesticides. The list of the residue limits for fenpropathrin in common baby supplement fruit and vegetable purees was made in Table 1 [4]. However, the safety limits for the use of fenpropathrin racemate have been only stipulated in these regulations, instead of the exact residue limits of fenpropathrin enantiomers.

At present, there are a variety of methods, such as GC [5, 6], CEC [7, 8], GC-MS [9, 10], LC-MS/MS [11–13], and HPLC [14–16], which are widely used in the field of chiral compound separation. Among them, the CEC chromatographic peak shape is good, but the analysis time is long; GC and GC-MS have high sensitivity and good resolution, but mainly analyze compounds with low boiling point and good thermal stability; HPLC features good resolution, but the organic reagents are consumed in large amounts; LC-MS/MS enjoys high accuracy, but the instruments used are expensive. SFC has received widespread attention as a high-efficiency chromatographic separation technology. The main mobile phase of this technique is supercritical carbon dioxide (CO_2), and the density and polarity of the mobile phase can be changed by adjusting the system back-pressure, chromatographic column temperature, and the ratio of organic solvents, so as to achieve precise control of the resolution of the target [17], which has obvious advantages in the application of chiral separation [18–21]. Nowadays, the application of SFC technology for the separation and residue determination of fenpropathrin enantiomers has not been reported.

In this study, the pretreatment method and the main parameters of instrumental chromatographic separation conditions for the fenpropathrin enantiomers in fruit and vegetable puree were optimized. An analytical method was firstly established for the rapid separation and determination of enantiomeric residues of fenpropathrin in the fruit and vegetable puree as supplementary food for infants by SFC technology. The samples of the represented fruit and vegetable puree as supplementary food for infants on the market were analyzed and determined. The method is efficient, rapid, reproducible, and environmentally friendly, enabling accurate analysis of pesticide enantiomers.

2 | MATERIALS AND METHODS

2.1 | Instruments, reagents, and standards

Acquity ultra-performance convergence chromatography (Acquity UPC^2) (Waters Company, USA, equipped with photo-diode array detector), desktop centrifuge (Thermo Company, USA), N-1210BV rotary evaporator (EYELA Tokyo Rikakikai Company, Japan), JJ500 electronic scale (G&G Company, USA), AE260 electronic scale (METTLER TOLEDO Company, Switzerland), WH-861 vortex mixer (Taicang Hualida Instrument Factory), Synergy185 ultrapure water meter (Millipore, USA), nitrogen blowing instrument (EYELA Tokyo Rikakikai Company, Japan), T18 basic grindomix (IKA Company, Germany), Microporous filter membrane (0.22 μm, organic phase), chromatographic column CHIRALPAK AD-3(150 × 3.0 mm^2, 3 μm, packed with amylose-tris (3,5-dimethylphenylcarbamate), OJ-H(100 × 4.6 mm^2, 5 μm, cellulose derivatives chiral column with spherical silica gel coated with chiral polymorphs (amylose or fiber derivatives)), IC(100 × 4.6 mm^2, 5 μm, cellulose-tris(3,5-dichlorophenyl carbamate) was immobilized through cross-linking mode in the surface of silicon) [22] (Daicel Chiral Technologies (China) Company), chromatographic column Acquity Trefoil CEL1(150 × 3.0 mm^2, 2.5 μm, cellulose-tris (3,5-dimethylphenylcarbamate)) (Waters Company, USA).

Isopropanol, acetone, methanol, heptane, ACN (chromatographically pure, Merck Company, Germany), sodium chloride (analytically pure, Tianjin Dingshengxin Chemical), hydrophile lipophile balance (HLB) column (Oasis, 200 mg, 6 mL), C_{18} column (CNW, 500 mg, 3 mL), Florisil column (CNW, 1 g, 6 mL), ultrapure water, other reagents used in the experiment are all analytically pure unless otherwise specified.

Standards: (−)-fenpropathrin, (+)-fenpropathrin (purity: ≥97%; Shanghai Chiralway Biotech).

2.2 | Preparation of standard solutions

Accurately weighted solid portions of two fenpropathrin enantiomer standards were dissolved in isopropanol to prepare 1.0 g/L of stock solution. These stock solutions were further diluted with heptane/isopropanol (9:1, v/v) to 1.0, 2.0, 4.0, 10.0, 20.0 mg/L to obtain calibration curves.

2.3 | Sample preparation

Note that, a 5 g sample was weighted (accurate to 0.01 g) and placed in a 50-mL plastic centrifuge tube, and then 20 mL ACN was added. The mixture was homogenized using a T18 basic grind mix. Note that, 3.0 g sodium chloride was added, for vortex mixing, 4000 r/min centrifugation for 5 min. The supernatant was transferred into a concentration bottle. The remaining residues were re-extracted once with 20 mL of ACN, as per the previous procedure. The organic layers were combined and evaporated to near dryness under vacuum at 40°C. The dried organic extract was reconstituted in 10 mL of ACN/water (1:1, v/v), for purification.

The solution to be purified was put in the activated HLB column, and after the sample was loaded, it was washed with 5 mL of water, then the lysate was discarded, drained, and eluted with 10 mL of acetone. The eluate was collected and dried by nitrogen in a water bath at 40°C until nearly dry. Constant volume was prepared with 1.0 mL heptane/isopropanol (9:1, v/v), and the constant volume solution was filtered through a 0.22 μm filter membrane for instrumental analysis.

2.4 | Instrumental analysis

The SFC analysis was performed using the CHIRALPAK AD-3 column. The elution gradient (eluent A: CO_2; eluent B: methanol) involved 3%B (initial), 3–10%B (0.2–0.21 min), and 10%B (0.21–2 min), and 10–3%B (2–2.5 min), and 3%B (2.5–3 min). The backpressure was 17.2 MPa. The detection wavelength was 230 nm. The flow rate was 1.5 mL/min. The column temperature was maintained at 31°C. The injection volume was 5 μL.

3 | RESULTS AND DISCUSSION

3.1 | Optimization of chromatographic condition

3.1.1 | Influence of detection wavelength

After scanning through the PDA detector, the UV spectrums of the standard solutions of fenpropathrin enantiomers were extracted from the chromatogram. There were obvious absorption peaks at 203, 230, and 275 nm, among which the absorption at 203 nm was the strongest and the sensitivity was relatively high, but the absorption of the interfering substances was also very strong at this wavelength.

The absorption at 230 nm was relatively high, and there are less interference peaks at the peaks of the fenpropathrin enantiomers. The absorption of the target compound was the lowest at 275 nm. For the detection of fenpropathrin compounds, it was more advantageous to use the 230 nm wavelength with higher absorbance and less impurity. Therefore, 230 nm was chosen as the detection wavelength in this experiment.

3.1.2 | Influence of chromatographic column

Chiral stationary phases based on amylose-tris(3,5-dimethylphenylcarbamate) and cellulose-tris(3,5-dimethylphenylcarbamate) are the two most widely used chromatographic stationary phases with good chiral recognition and splitting ability, complementing each other in terms of chiral recognition ability [23]. In this experiment, four chiral separation chromatographic columns, namely CHIRALPAK AD-3, CHIRALPAK OJ-H, CHIRALPAK IC, and Acquity Trefoil CEL1, were selected to investigate the resolution of the two fenpropathrin enantiomers. The results showed that the chromatographic peaks of the two fenpropathrin enantiomers completely overlapped when separated by IC and CEL1 chiral chromatographic columns. When the OJ-H chiral column was used for separation, the chromatographic peaks of the two fenpropathrin enantiomers could achieve a certain separation, but the separation factor is poor, and the peak shape of the chromatographic peak is broadened obviously. In contrast, the separation factor was good and

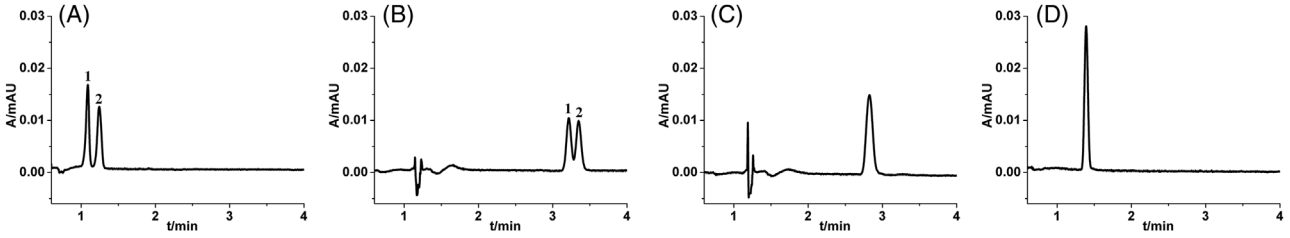

FIGURE 2 Effect of different chromatographic columns on the separation of (−)-fenpropathrin and (+)-fenpropathrin (co-solvent: methanol, backpressure: 13.8 MPa, chromatographic column temperature: 35°C). (A) AD-3, (B) OJ-H, (C) IC, and (D) CEL1. Peak 1: (−)-Fenpropathrin; Peak 2: (+)-Fenpropathrin

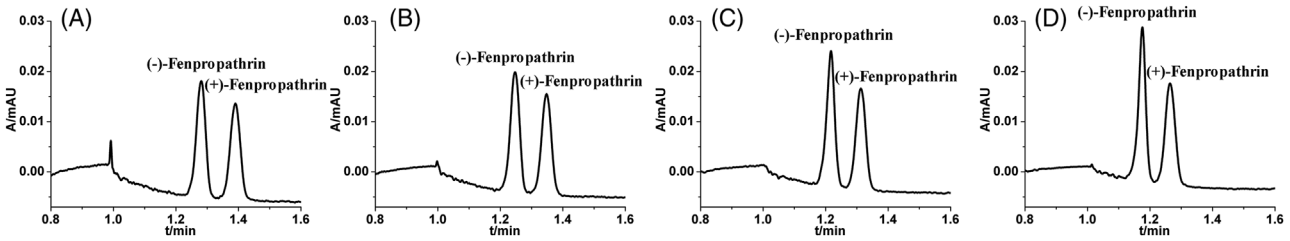

FIGURE 3 Effect of different system backpressure on the separation of (−)-fenpropathrin and (+)-fenpropathrin (co-solvent: methanol, chromatographic column temperature: 31°C). (A) 10.3 MPa, (B) 12.1 MPa, (C) 13.8 MPa, and (D) 17.2 MPa

the chromatographic peak shape was sharp when the AD-3 chiral column was used for separation (see Figure 2). Therefore, the AD-3 chiral column was chosen for the separation of fenpropathrin enantiomers in this experiment.

3.1.3 | Influence of backpressure

With supercritical CO_2 as the mobile phase, SFC can effectually alter the viscosity and density of the mobile phase by controlling the system backpressure during the experiment, thereby adjusting the dissolution and elution strength of the mobile phase. The viscosity and density of the mobile phase will increase as the system backpressure increases. As the CO_2 pressure transcends 7.38 MPa and the chromatographic column temperature exceeds 31°C, CO_2 will reach the supercritical state. Therefore, the effects of the four system backpressures of 10.3, 12.1, 13.8, and 17.2 MPa on the separation of two fenpropathrin enantiomers were investigated in this experiment. As shown in Figure 3, when the system backpressure was gradually increased in the experiment, the retention time of the compound was reduced, and the peak shape became sharper. Under the four conditions, the two fenpropathrin enantiomers reached the best chromatographic peak shape when the system backpressure was 17.2 MPa. Considering the highest recommended system backpressure of the AD-3 chiral column, 17.2 MPa was selected as the best one in this study.

3.1.4 | Influence of chromatographic column temperature

The viscosity of the mobile phase gradually decreased, its density reduced, and the elution ability on the target also weakened, and the retention time was lengthened, with the rise of chromatographic column temperature. Considering that the maximum operating temperature of the CHIRALPAK AD-3 chiral column is 40°C, CO_2 would enter the supercritical state when the temperature is over 31°C and the backpressure exceeds 7.38 MPa. Therefore the effect of the column temperature in the range of 31–40°C on the peak shape and separation of the target compounds was investigated in the experiment. Results indicated that the separation factor of the two fenpropathrin enantiomers was poor when the column temperature was 35–40°C. When the column temperature was 31°C, the two kinds of fenpropathrin enantiomers were well separated and achieved a good baseline separation within 1.4 min. Therefore, 31°C was selected as the optimal temperature.

3.1.5 | Influence of constant volume dilution solvent

The experiment studied the impacts of five common constant volume dilution solvents for SFC such as methanol, ethanol, ACN, isopropanol, and heptane on the separation of two kinds of fenpropathrin enantiomers. The results

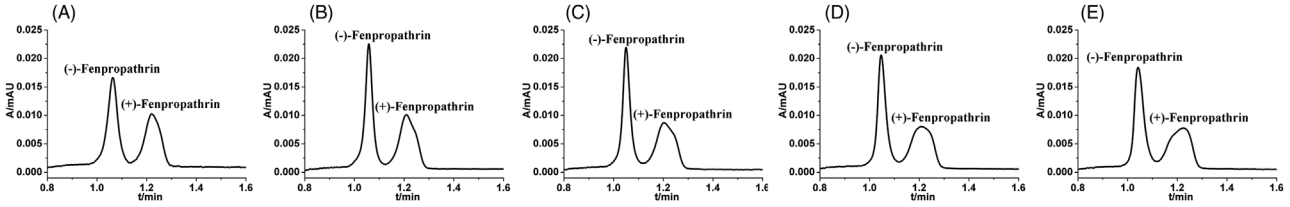

FIGURE 4 Effect of different constant volume dilution solvents on the separation of (−)-fenpropathrin and (+)-fenpropathrin (co-solvent: methanol, backpressure:17.2 MPa, chromatographic column temperature: 31°C). (a) heptane/isopropanol (9:1, v/v); (b) heptane/isopropanol (8:2, v/v); (c) heptane/isopropanol (7:3, v/v);(d) heptane/isopropanol (6:4, v/v);(e) heptane/isopropanol (5:5, v/v)

showed that the best peak shape was obtained when heptane was used. Considering the poor solubility of heptane when heptane was used as the constant volume solution in the subsequent sample pretreatment step, thus reducing the recovery of fenpropathrin enantiomers, a mixture of heptane and isopropanol was used in the experiment. At the same time, this experiment investigated the effect of different ratios of heptane and isopropanol mixed solutions on the chromatographic peak shape of fenpropathrin. The peak shape of (−)-fenpropathrin was gradually sharpened and that of (+)-fenpropathrin gradually deteriorated as the proportion of isopropanol increased (see Figure 4). Considering the peak shape and solubility of the target compounds, heptane/isopropanol (9:1, v/v) was finally determined as the constant volume dilution solvent for this experiment.

3.2 | Optimization of pretreatment procedure

The experiments were conducted to compare the purification effect of different kinds of SPE columns such as HLB [24–26], C_{18} [27], and Florisil [28] on the extraction solution of fruit and vegetable puree samples. Two kinds of fenpropathrin enantiomer standard solutions were added to the fruit and vegetable puree samples without fenpropathrin, followed by the homogeneous extraction with ACN two times. The extracts were concentrated, dried, and re-dissolved with heptane/isopropanol (9:1, v/v), and then cleaned up by three different kinds of SPE columns, respectively. The experimental results showed that when the Florisil column was used for the purification, there were interference peaks for (−)-fenpropathrin and the recovery was 29.2%. When the HLB column was used for the purification, the average recoveries of both fenpropathrin enantiomers could reach more than 95.5%. The reproducibility of sample determination using the HLB column was better than that of the C_{18} column, which was consistent with the phenomenon reported in the literature [24]. Therefore, the HLB column was finally chosen as the purification column for the experiment.

3.3 | Linearity and LOQs

The enantiomeric standard solutions of 1.0, 2.0, 4.0, 10.0, and 20.0 mg/L in Section 4 were selected and measured according to the optimized chromatographic conditions, and the standard curves were plotted, with the corresponding peak areas as the vertical coordinates (y) and the mass concentrations of the standard solutions as the horizontal coordinates (x). The linear regression was performed in the experiment. The determinations were performed in terms of the optimized method by adding standards into the three kinds of fruit and vegetable puree samples without fenpropathrin. The LOQs (S/N = 10) of two kinds of fenpropathrin enantiomers were both 0.2 mg/kg for all samples. The linearity was good in the linear range of 1.0–20.0 mg/L and the correlation coefficients (r) were all greater than 0.9992.

3.4 | Precision and accuracy

The spiked recoveries and the precision of the method were determined by adding the standard solution to the blank samples of fruit and vegetable puree without fenpropathrin. The spiked levels of (−)-fenpropathrin and (+)-fenpropathrin were 0.2, 0.4, and 2.0 mg/kg, respectively, and the spiked recoveries and RSDs were calculated six times in parallel. The recoveries of the two target compounds ranged from 80.6 to 105% and the RSD values (n = 6) ranged from 2.6 to 7.7% (see Table 2). The recovery and precision met the requirements of SANTE/12682/2019 [29], which can meet the analytical requirements of apple puree, strawberry puree, and tomato puree samples for daily analysis.

3.5 | Real samples analysis

In order to examine the practicality of the method, the two kinds of fenpropathrin enantiomers were tested in 30 samples of commercially available apple, strawberry, and tomato purees randomly selected by the developed

TABLE 2 Spiked recoveries and RSDs of two kinds of fenpropathrin enantiomers in apple puree, strawberry puree, and tomato puree matrixes ($n = 6$)

Fenpropathrin enantiomer	Spiked (mg/kg)	Apple puree		Strawberry puree		Tomato puree	
		Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
(−)-Fenpropathrin	0.2	80.6–95.2	5.8	85.8–104	6.7	85.4–103	6.8
	0.4	87.4–97.9	6.7	81.4–98.8	5.7	85.4–101	5.0
	2.0	86.4–96.4	2.9	82.6–98.2	4.6	82.6–94.5	2.6
(+)-Fenpropathrin	0.2	81.7–98.3	6.7	82.8–105	7.5	84.8–105	7.7
	0.4	81.4–94.3	5.6	83.5–103	5.9	81.0–97.8	6.5
	2.0	85.8–96.4	3.5	84.2–99.6	4.6	84.3–96.8	4.0

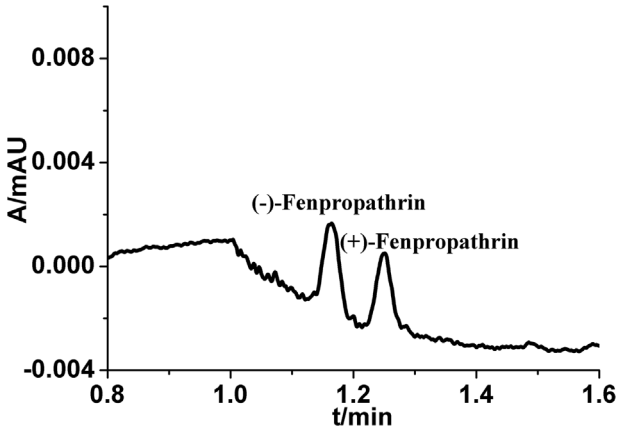

FIGURE 5 Chromatograms of the actual positive tomato puree sample (co-solvent: methanol, backpressure: 17.2 MPa, chromatographic column temperature: 31°C)

method. The results exhibited that two kinds of fenpropathrin enantiomers were detected in one tomato puree sample. The two fenpropathrin enantiomers were effectively separated within 1.4 min, with a separation degree of 1.7, meeting the requirement of $R \geq 1.5$ for complete separation [30] (see Figure 5). The analysis time was shorter, and the separation degree was higher than the method reported in the literature [1, 2] for the separation by the HPLC method. In accordance with the retention time order of the chromatographic peaks, the two fenpropathrin enantiomers were in order as follows: (−)-fenpropathrin, (+)-fenpropathrin. Based on the standard curve plotted above, the contents of (−)-fenpropathrin and (+)-fenpropathrin were calculated by the external standard quantification method, in which the content of (−)-fenpropathrin was 0.21 mg/kg and that of (+)-fenpropathrin was 0.24 mg/kg, respectively.

4 | CONCLUDING REMARKS

In this paper, a method was developed for the separation and determination of fenpropathrin enantiomers residues in the represented fruit and vegetable puree (such as apple puree, strawberry puree, and tomato puree) as supplementary food for infants by SFC technology. The samples were extracted with ACN, cleaned up by HLB column, separated by CHIRALPAK AD-3 chiral column, eluted with supercritical CO_2-methanol as mobile phase gradient, and quantified by external standard method.

The LOQs were both 0.2 mg/kg, the recoveries ranged from 80.6 to 105%, and the RSD reached 2.6–7.7%. The method was adopted to determine the content of fenpropathrin enantiomer residues in 30 market-purchased fruit and vegetable puree samples, and the detection amount ranged from 0.21 to 0.24 mg/kg.

ACKNOWLEDGMENTS

This work was supported by the National Program on Key Research Project of China (2019YFE0103900).

CONFLICT OF INTEREST

The author has declared no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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REFERENCES

1. Zhu MN, Li ZY, Li QL, Cao YH, Chen FN. Study on the enantioselective degradation of fenpropathrin in soil. Jiangsu Agric Sci. 2011;39:481–3.
2. Gao RY, Zhu LY, Chen ZY. Separation of pyrethroid enantiomers of fenprapathrin and fluvalinate by chiral high performance liquid chromatography. Pesticides 1998;37:22–4.
3. Dalian Institute of Chemical Physics. Chiral synthesis and industrialization of s-cyanohydrin and s-cypermethrin. http://www.dicp.ac.cn/kycg_1/yyyjcg/201809/t20180906_5064992.html

4. China Technical Trade Measures Network. Pesticide and veterinary drug residues. http://www.tbtsps.cn/page/trade/Foodlimitaddlive.action?pageTypes=2&type=2
5. Shin DW, Ko BJ, Cheong JC, Lee W, Kim S, Kim JY. Impurity profiling and chemometric analysis of methamphetamine seizures in Korea. Anal Sci Technol. 2020;33:98–107.
6. Motoko W, Hidehiko W, Devenie RA, Wolfgang E, Heinz EK. Analytical and sensory characterization of the stereoisomers of 3-mercaptocycloalkanones and 3-mercaptocycloalkanols. J Agr Food Chem. 2020;68:7184–93.
7. Balint A, Cârje AG, Muntean DL, Imre S. Research Article. The influence of some parameters on chiral separation of ibuprofen by high-performance liquid chromatography and capillary electrophoresis. Acta Medica Marisiensis. 2017;63:36–40
8. Storch J, Kalíková K, Tesařová E, Maier V, Vacek J. Development of separation methods for the chiral resolution of hexahelicenes. J Chromatogr A. 2016;1476:130–4.
9. Duell AK, Kerber PJ, Luo WT, Peyton DH. Determination of (R)-(+)- and (S)-(−)-nicotine chirality in puff bar e-liquids by ^{1}H NMR spectroscopy, polarimetry, and gas chromatography-mass spectrometry. Chem Res Toxicol. 2021;34:1718–20.
10. Casilli A, Decorzant E, Jaquier A, Delort E. Multidimensional gas chromatography hyphenated to mass spectrometry and olfactometry for the volatile analysis of citrus hybrid peel extract. J Chromatogr A. 2014;1373:169–78.
11. KarakkaKal AK, Karatt TK, Philip M, Meissir S, Nalakath J. Separation and determination of the enantiomeric levamisole and dexamisole in equine plasma samples using chiral polysaccharide column/LC-MS/MS. Curr Anal Chem. 2020;16:761–7.
12. Yoshiyuki O, Masahito T, Togo S, Toru A. LC-MS/MS and chiroptical spectroscopic analyses of multidimensional metabolic systems of chiral thalidomide and its derivatives. Chirality 2017;29:282–93.
13. Singh SP, Dwivedi N, Raju KSR, Taneja I, Wahajuddin M. Validation of a Rapid and Sensitive UPLC-MS-MS method coupled with protein precipitation for the simultaneous determination of seven pyrethroids in 100 μl of rat plasma by ssing ammonium adduct as precursor ion. J Anal Toxicol. 2016;40:213–21.
14. Gurupadayya B, Reddy M, Prabhakar P, Raikar P, Mandal SP. Chiral separation of oxomemazine enantiomers by HPLC technique and enantiomeric separation mechanism via docking studies. Curr Pharm Anal. 2021;17:222–30.
15. Suthar SK, Rauscher AÁ, Winternitz M, Gyimesi M, Málnási CA. Chiral HPLC separation of enantiomeric blebbistatin derivatives and racemization analysis in vertebrate tissues. J Pharmaceut Biomed. 2021;204:114–246.
16. Jing X, Huang X, Wang HH, Xue HY, Wu BQ, Wang XW, Jia LY. Popping candy-assisted dispersive liquid-liquid microextraction for enantioselective determination of prothioconazole and its chiral metabolite in water, beer, Baijiu, and vinegar samples by HPLC. Food Chem. 2021;348:129–47.
17. Antpedia. Ultra-performance convergence chromatography (UPC^2): new categories of chromatography have empowered scientists with new imaginations. https://www.antpedia.com/index.php?action-viewnews-itemid-212253-php-1
18. Zhang WH, Xie W, Hou JB, Chen QK, Li SM, Zhu ZL, Zou XQ. Chiral separation of six triazole pesticide enantiomers by ultra-performance convergence chromatography and residue determination in cucumber. Chin J Chromatogr. 2019;37:1356–62
19. Jiang H, Yang L, Xing XD, Yan ML, Guo XY, Yang BY, Wang QH, Kuang HX. Development of an analytical method for separation of phenolic acids by ultry-performance convergence chromatography(UPC^2) using a column packed with a sub-2-μm particle. J Pharmaceut Biomed. 2018;153:117–25.
20. Yu WS, Liu X, Zhang YZ, Lin YN, Qiu J, Kong FY. Simultaneous determination of pigments in tea by ultra-performance convergence chromatography (UPC^2). Anal Lett. 2020;53:1654–66.
21. Zhang WH, Xie W, Hou JB, Hu XL, Wang P, Zhang YQ, Xu DM. Analytical research on the separation and residue of chiral pesticide triadimenol in fruit and vegetable puree. J Sep Sci. 2021;44:3516–23.
22. Francotte E, Huynh D. Immobilization of 3,5-dimethylphenyl carbamate of cellulose and amylose on silica by photochemical and thermal radical processes. Chirality 2022;34:711–31.
23. Okamoto Y, Yashima E. Polysaccharide derivatives for chromatographic separation of enantiomers. Angew Chem Int Ed. 1998;37:1020–43.
24. Dong SY. Study on extraction, clean-up and ultra-performance liquid chromatographic analysis of fluvalinateand flumethriin residues in royal jelly. Food Sci. 2009;30:272–4.
25. Wu F, Shao AM, Xu XP. Simultaneous determination of atrazine, dichloropermethrin, cypermethrin, mepermethrin and permethrin in drinking water by HPLC. Mod Prevent Med. 2020;47:3619–22.
26. Luo Y. Determination of pythiril, fenvalerate, fenpropathrin and pentachloronitrobenzene residues in soil by solid phase extraction GC-ECD method. Chem Eng. 2020;299:28–30.
27. Li MQ, An WJ, Li J, Wang L, Zhuang J, He ML. Determination of 19 pesticides residues in eggs by gas chromatography with solid phase extraction. J Instr Anal. 2020;39:520–5.
28. Li SL. Determination of putrescine, pentachloronitrobenzene and cypermethrin residues in Vaquita by solid phase extraction-GC-ECD. Light Industry Science and Technology. 2020;36:125–6.
29. SANTE/12682/2019 Guidance document on analytical quality control and method validation procedures for pesticide residues analysis in food and feed.
30. Baipharm. Pharmacopoeia of the People's Republic of China. Volume I. 2020.

How to cite this article: Zhang W-h, Xu D-m, Hou J-b, Zhang Y-q, Zhu Z-l, Mao R-y, Qui H, Xie W, Shen W-j, Yi X-h. Research method of rapid determination of chiral pesticide fenpropathrin enantiomers in fruit and vegetable puree by supercritical fluid chromatography. J Sep Sci. 2022;45:2717–2723.
https://doi.org/10.1002/jssc.202200147