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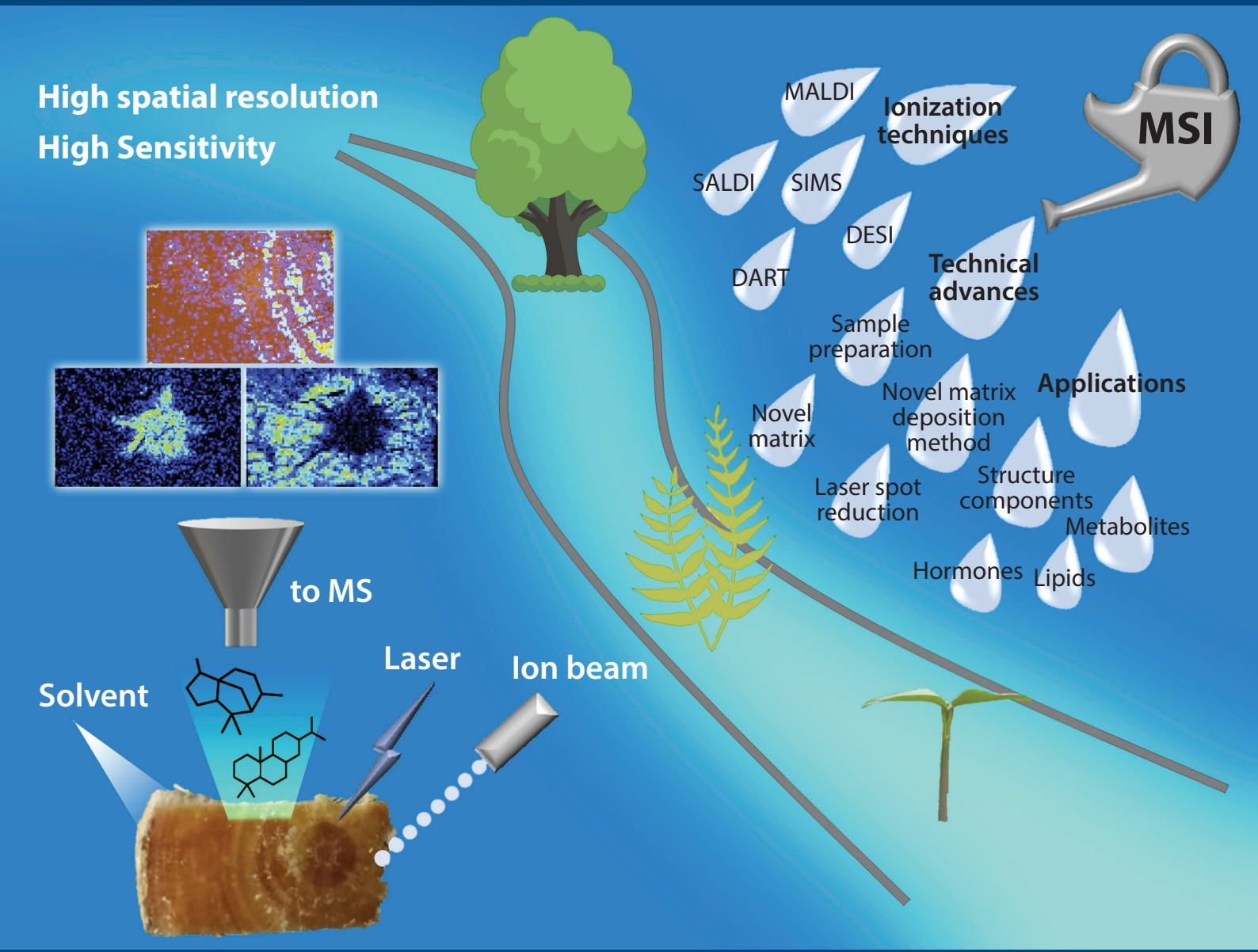

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RESEARCH ARTICLE

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Analytical research on the separation and residue of chiral pesticide triadimenol in fruit and vegetable puree

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In this paper, a method for the separation of triadimenol stereoisomers using ultra-performance convergence chromatography and an analytical method for the determination of triadimenol stereoisomer residues in pumpkin puree, apple puree, and tomato puree as a supplement for infants are established. Test samples were extracted with acetonitrile and successively purified with graphitized carbon black and Florisil column. Afterward, Acquity Trefoil AMY1 column was adopted for chiral separation of chromatographic column, and gradient elute was carried out with supercritical carbon dioxide-methanol as the mobile phase and with external standard method for quantitation. Results showed that the linearly dependent coefficient of the four kinds of triadimenol stereoisomers within 1.0 and 50 mg/L was greater than 0.9997, and the limit of quantitation of the four kinds of triadimenol stereoisomers was 0.05 mg/kg. Recovery experiment was carried out within 0.05 and 1.0 mg/kg scope, the recoveries were 81.0–107%, and the relative standard deviation was 2.3–7.6%. This method implemented the separation of triadimenol stereoisomers and its residue test in pumpkin puree, apple puree, and tomato puree as a supplement for infants, and it can provide reliable technical support for the analysis of pesticide residue and assessment of product quality.

KEYWORDS
chiral separation, puree, stereoisomers, triadimenol, ultra-performance convergence chromatography

1 | INTRODUCTION

Triazole is a kind of organic fungicides, which is one of the top 10 pesticides in its usage (https://www.chyxx.com/industry/201707/541622.html) [1]. Triadimenol, whose chemical name is 1-(4-chlorophenoxy)−3, 3-dimethyl−1-(1H-1, 2, 4-triazol-l-yl)−2-butanol, is a kind of important triazole fungicide, featuring broad-spectrum bactericidal activity, and it is usually used for the disease control of vegetables and fruits. Its pollution to the environment and food has also attracted extensive attention [2]. Research shows that triadimenol has two chiral carbon atoms and two stereoisomers, in which the stereoisomer in (+)-(*R,S*)-triadimenol and (-)-(*S,R*)-triadimenol structure is called triadimenol A, and the other in (-)-(*S,S*)- triadimenol and (+)-(*R,R*)- triadimenol structure is called triadimenol B [3] (see Figure 1).

Different triadimenol stereoisomers differ greatly in bioactivity. Specifically, *SR* of triadimenol stereoisomer has better pesticide effect and lower toxicity than *RS*, *SS* and

Article Related Abbreviations: Carb/NH_2, graphitized carbon black column-amino; UPC^2, ultra-performance convergence chromatography

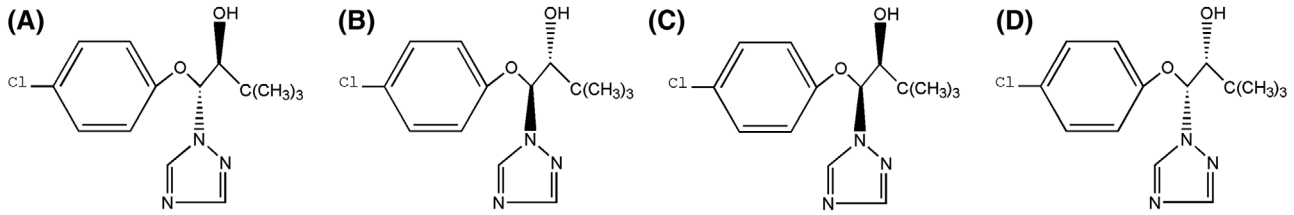

FIGURE 1 The chemical structures of different triadimenol stereoisomers. (a) (+)-(*R*,*S*)-Triadimenol; (b). (-)-(*S*,*R*)-triadimenol; (c) (-)-(*S*,*S*)-triadimenol; and (d) (+)-(*R*,*R*)-triadimenol

TABLE 1 The maximum residue limits of triadimenol in pumpkin, apple, and tomato matrixes

Matrix	European union (mg/kg)	Japan (mg/kg)	China (mg/kg)
Pumpkin	0.2	2	0.2
Apple	0.2	0.5	1
Tomato	0.3	0.5	1

RR of triadimenol stereoisomer [4, 5]. In order to standardize the use of such pesticide, many countries have established maximum residue limit standard of triadimenol in fruits and vegetables. Table 1 lists the residue limit of triadimenol in common supplementary fruit and vegetable puree for babies. But these laws and regulations just stipulate the safety limit for the use of triadimenol raceme, rather than the accurate residue limit of triadimenol stereoisomers.

Currently, GC [6,7], GC-MS [8,9], CEC [10,11], HPLC [12,13], LC-MS/MS [14,15], and other methods play an important role in the separation of chiral compound. Dong et al. [3,5] and Wang et al. [16] have reported that triadimenol stereoisomers were successfully separated on Chiral column using HPLC method. But these methods usually have several problems, such as long analysis time, poor chromatographic peak shape, unsatisfactory degree of separation, huge consumption of organic reagent, and so on. In recent years, ultra-performance convergence chromatography (UPC^2) has been applied in chiral separation for its remarkable advantages, since it takes supercritical carbon dioxide and a little organic solvent (acetonitrile, methanol, isopropyl alcohol, etc.) as the mobile phase, and obtains the required systemic resolution through accurately regulating the ratio of mobile phase, chromatographic column temperature, and system back pressure, then accurately regulate the retention time and the degree of separation of chiral compound. UPC^2 integrates supercritical fluid chromatography and UHPLC [17]. Recently, several studies on the application of supercritical fluid chromatography and UPC^2 in separating triazole fungicides [18–23], acaricide cyflumetofen [24], neonicotinoid insecticides [25], and organophosphorus insecticide [26] have been reported.

In this research, a method for the separation of triadimenol stereoisomers by using UPC^2 and an analytical method for the determination of triadimenol stereoisomer residues in pumpkin puree, apple puree, and tomato puree as a supplement for infants are established. The experiment observed the stability of derivatives of four kinds of triadimenol stereoisomers, optimized the pre-treatment method of triadimenol stereoisomers in fruit and vegetable puree as well as main parameters like chromatographic separation conditions, analyzed and tested main supplementary fruit and vegetable puree samples for babies with the optimization method. Featuring fast analysis speed, good separation effect, and low consumption of organic solvent, etc., this method can provide guidance for the chiral analysis of other pesticides.

2 | MATERIALS AND METHODS

2.1 | Instruments, materials, and reagents

UPC^2 (Waters Corporation, USA), desk centrifuge (Thermo Company, USA), R215 rotary evaporators (Buchi Company, Switzerland), AE260 electronic balance (Mettler Company, Switzerland), MS2 scroll mixer (Shanghai Yida Instrument Factory), ultrapure water purification system (Elga Company, UK), microporous filtration membrane (0.22 μm, organic phase), and nitrogen blowing instrument (RKC INSTRUMENT, Japan).

Hexane, acetone and acetonitrile (chromatographically pure, Scharlau Company, Spain), anhydrous sodium sulfate (analytically pure, 450°C calcination, Shanghai No.4 Reagent & H.V. Chemical), Florisil adsorbent (superior purity, 100–200 mesh; Sinopharm Chemical Reagent, calcined at 650°C for 24 h, cooled to room temperature, and then deactivated with 2% water for standby application); GCB (graphitized carbon black), PSA (*N*-propyl ethylenediamine), and C_{18} powder (superior purity, Shanghai Anpel Scientific Instrument); anhydrous magnesium sulfate (analytical purity, Xilong Science); self-made Florisil column (10 g, 60 mL); and Florisil column (CNW, 5 g, 60 mL), graphitized carbon black column-amino

(Carb/NH_2) composite column (Agela, 500 mg, 6 mL), ultrapure water, other experimental reagents are all analytically pure unless otherwise specified.

Standards: Triadimenol raceme (purity ≥ 99%; Dr Ehrenstorfer, Germany). Triadimenol stereoisomer: (+)-(*R*,*S*)- triadimenol, (-)-(*S*,*R*)- triadimenol, (-)-(*S*,*S*)- triadimenol, (+)-(*R*,*R*)- triadimenol (purity ≥ 95%; Shanghai Chiralway Biotech).

2.2 | Stock and preparation of working standard solutions

Standard stock raceme solution (1.0 g/L): An appropriate amount of the above-stated standard triadimenol raceme was weighed and dissolved in acetonitrile in a constant volume to prepare 1.0 g/L standard stock solution.

Stock solution of four kinds of triadimenol stereoisomers (1.0 g/L): An appropriate amount of the above-stated standard triadimenol stereoisomer was weighted separately and dissolved in acetonitrile in a constant volume to prepare 1.0 g/L standard stock solution.

Calibration mixed standards of four kinds of triadimenol stereoisomers: Four kinds of triadimenol stereoisomers calibration mixed standards were prepared at concentrations of 1.0, 2.0, 4.0, 10, 20, and 50 mg/L before use.

2.3 | Preparation of the sample

Note that 20 g test sample was weighted (accurate to 0.01 g) and placed in a 100-mL polypropylene centrifuge tube, and then 40 mL acetonitrile was added; for homogeneous extraction, 3.0 g sodium chloride was added; for vortex mixing, 4000 r/min centrifugation for 5 min. Supernatant was taken and placed in another 100-mL polypropylene centrifuge tube. The extraction was repeated once, and the supernatant was combined twice, for purification.

The above extract was moved to a 100-mL polypropylene centrifuge tube which contained 1.0 g graphitized carbon black powder. After 1 min of vortex mixing and 5 min of 8500 r/min centrifugation, the supernatant was moved to a concentration bottle, and concentrated with a rotary evaporator till it was nearly dry. The residue was redissolved with 50 mL acetone/hexane (1:1, *v*/*v*) mixture for three times. The redissolved solution was moved to a glass tube with 10 g 2% dehydrated Florisil, and the sample was loaded and purified solution was received directly and then concentrated with a rotary evaporator till it was nearly dry. Constant volume was prepared with 1.0 mL acetonitrile, and the constant volume solution was filtered through 0.22 μm filter membrane.

2.4 | Chromatographic conditions

The UPC^2 analysis was performed using an Acquity Trefoil AMY1 column. The elution gradient (eluent A: carbon dioxide (CO_2); eluent B: methanol) involved 10%B (initial), 10–30% B (2-2.5 min), and 30% B (2.5-5 min), and 30–10% B (5-5.5 min), and 10% B (5.5-6.5 min). The backpressure was set to 13.8 MPa. The flow rate was 2.0 mL/min, and the injection volume was 10 μL. The column temperature was maintained at 40°C. The detected wavelength was 220 nm.

3 | RESULTS AND DISCUSSION

3.1 | Stability study of triadimenol stereoisomer standard

This experiment observed the stability of the four kinds of 10 mg/L triadimenol stereoisomer standard solution kept for 1, 3, 5, 7, 14, 30, and 60 days at −18 °C. Freshly prepared standard solutions of triadimenol stereoisomer (10 mg/L) were compared with the content of 10 mg/L triadimenol stereoisomers kept for 1, 3, 5, 7, 14, 30, and 60 days, respectively, at −18°C, with the freshly prepared standard solution as 100%, and changes of triadimenol stereoisomer standard solution smaller than 10% as the benchmark. Results showed that content of the four kinds of triadimenol stereoisomers dropped gradually (see Supporting Information Figure S1), in which the content dropped by more than 25% after being kept for 60 days at −18°C, and more than 22% after being kept for 30 days, and more than 12% after being kept for 14 days, but less than 10% after being kept for 7 days. The experimental results indicated that the four kinds of triadimenol stereoisomers were relatively stable in 7 days.

3.2 | Optimization of chromatographic condition

3.2.1 | Influence of cosolvent

Considering that triadimenol is soluble in water and methanol and the mobile phase of UPC^2 system is mainly nonpolar CO_2, and a little cosolvent should be added to increase the efficiency of separation, in order to strengthen the polarity of mobile phase and guarantee the elution of polar components. The experiment studied the impacts of different cosolvents such as methanol, isopropanol:ethanol (1:1, v/v), isopropanol:hexane (1:3, v/v), and so on on the separation of four kinds of triadimenol stereoisomers (see Figure 2). Results showed that

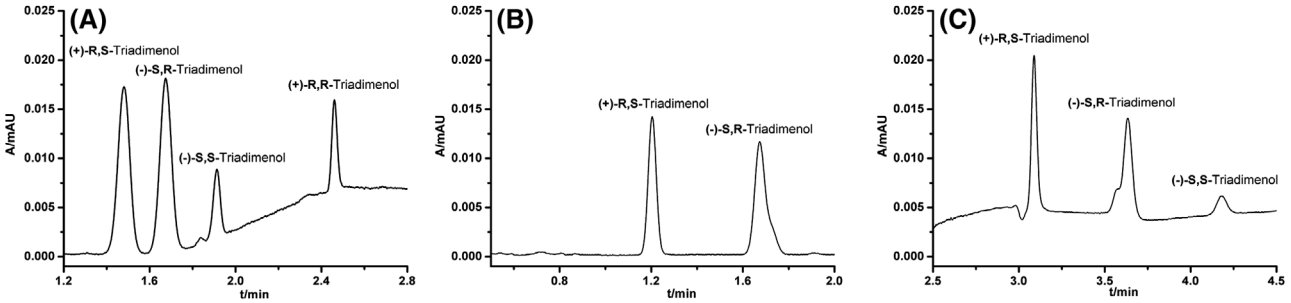

FIGURE 2 Effect of different organic solvent mobile phases on separation of four kinds of triadimenol stereoisomers. (a) Methanol; (b) isopropanol–ethanol (1:1, v/v); and (c) isopropanol–hexane (1:3, v/v)

the four kinds of triadimenol stereoisomers failed to show all peaks, and some chromatographic peaks overlapped and widened remarkably, when isopropanol–ethanol (1:1, v/v), and isopropanol–hexane (1:3, v/v) were employed for cosolvents. When methanol was taken as the cosolvent, the chromatographic peaks of the four kinds of triadimenol stereoisomers were completely separated within 3 min. Therefore, methanol was adopted as cosolvent in this experiment.

3.2.2 | Influence of backpressure

With supercritical CO_2 as the mobile phase, UPC[2] can effectively change the density of CO_2 by adjusting the system backpressure and column temperature, thus change its dissolving capacity, elution power, and selectivity of substance. CO_2 would enter the supercritical state when the column temperature is over 31°C and the pressure exceeds 7.38 MPa. The influence of different back pressure ranging between 10.3, 13.8, and 17.2 MPa on the separation effect of the four kinds of triadimenol stereoisomers was observed, with methanol as cosolvent and the column temperature was 40°C. Results showed that the retention time of the target compound was shortened with the increase of system backpressure. Under three conditions, the chromatographic peak shape and degree of separation of the four kinds of triadimenol stereoisomer were the best, when the back pressure was 13.8 MPa. Considering the retention time, peak shape, and system pressure, the backpressure was set to be 13.8 MPa in this research.

3.2.3 | Influence of chromatographic column temperature

The research found that the viscosity of CO_2 supercritical liquid reduced, and its elution power on the target also weakened, and the retention time was lengthened, with the rise of chromatographic column temperature. Considering that the highest recommended operating temperature of Acquity Trefoil AMY1 chiral chromatographic column is 40°C, and CO_2 would enter the supercritical state when the temperature is over 31 °C and the backpressure exceeds 7.38 MPa, therefore this experiment observed the influence of chromatographic temperature ranging between 31 and 40°C on the separation of triadimenol stereoisomers. Results showed that the retention time of target compound extended gradually with the rise of the column temperature. When the column temperature was 31 and 35°C, the degree of separation of the four kinds of triadimenol stereoisomers was unsatisfactory. When the temperature rose to 40°C, the peak shape of the four kinds of triadimenol stereoisomers was good, and completely separated within 3 min. Therefore, 40°C was selected as the optimal temperature.

Figure 2a shows the separation of four kinds of triadimenol stereoisomers when methanol was taken as the cosolvent, the back pressure was 13.8 MPa, and the chromatographic column temperature was 40°C. The four kinds of triadimenol stereoisomers could be separated effectively in 3 min, and according to the retention time of chromatographic peak, and it was followed by (+)-(*R*,*S*)-triadimenol, (-)-(*S*,*R*)- triadimenol, (-)-(*S*,*S*)- triadimenol, and (+)-(*R*,*R*)- triadimenol.

3.3 | Optimization of pretreatment conditions

3.3.1 | Optimization of extraction reagent

There are some literatures about the determination of triadimenol pesticide in food, and triadimenol pesticide was extracted with 70% ethanol [27], methanol [28], acetonitrile [29, 30], and dichloromethane [31], respectively. This paper examined the extraction effect of these reagents. Results showed that when methanol, 70% ethanol, and dichloromethane were taken as the extraction reagent, respectively, the chromatogram of (+)-(*R*,*R*)-triadimenol

showed matrix interference peak, and dichloromethane had high toxicity. When acetonitrile was taken as the extraction reagent, there were few matrix interference peaks in the chromatogram of the four kinds of triadimenol stereoisomers. Consequently, acetonitrile was adopted in this research as the extraction solvent.

3.3.2 | Optimization of purification conditions

LLE purification, SPE purification, QuEChERS, and other purification methods are usually adopted for the purification of fruit and vegetable puree samples to eliminate the background interference brought by such substance as pigment, fat and fatty acid, vitamins and carbohydrates, and to improve the accuracy of detection results. This paper investigated that the purification effect of two kinds of SPE columns, namely Florisil column and Carb/NH_2 composite column. Four kinds of triadimenol stereoisomer standard solution were added into the pumpkin puree, apple puree, and tomato puree samples free from triadimenol, homogeneous extraction was conducted twice with acetonitrile, and the extracted solution was purified with graphitized carbon black, following by the purification with two different kinds of SPE columns, respectively. The experiment found that when Carb/NH_2 composite column was adopted for purification, (-)-(*S,S*)- triadimenol had matrix interference peak, and it was unable to conduct quantitative determination. Florisil column was adopted for purification that can remove the matrix interference to the target peak [32], and the mean recovery rate of the target analytes was 95.0%.

This experiment also observed the purification effect of self-made Florisil column and commercial Florisil column on the extraction solution of fruit and vegetable puree samples. Florisil column usually requires inactivation with water, while self-made Florisil column makes it easier to control the water content of Florisil. Moreover, commercial Florisil column is packaged, and each Florisil column has different water content, which results in distinct activity and poor repeatability in sample detection. In addition, Florisil column sold in the market is mainly 1, 2 or 5 g, and 10 g is rarely sold. In the experiment, the fruit and vegetable puree was concentrated ten times, with a high impurity interference, and the column capacity of commercial Florisil column (1, 2, 5 g) failed to meet the purification requirements. Therefore, 10 g Florisil column was required in this experiment. Upon overall consideration, this experiment eventually adopted self-made 10 g Florisil column to purify the extraction solution of fruit and vegetable puree samples.

3.4 | Method validation

3.4.1 | Linearity of calibration standards

Mixed standard solution of the four kinds of triadimenol stereoisomers was tested according to the above-stated chromatographic conditions, and standard curve was drawn with the peak area of standard substance (y) as the y-coordinates and the corresponding quality concentration (x) as the x-coordinates. The linear equation and correlation coefficient of the four kinds of triadimenol stereoisomers were calculated. Results showed that the four kinds of triadimenol stereoisomers presented good linear relationship within 1.0 and 50 mg/L, and the correlation coefficient (r) was greater than 0.9997 (see Table 2). Tests were conducted according to this method by adding standard substance into the fruit and vegetable puree samples free from triadimenol, and the limit of quantitation was calculated as lowest concentration level validated with satisfactory recoveries between 80 and 120% with ten times the relative standard deviation. As a result, the LOD for the four kinds of triadimenol stereoisomers was 0.05 mg/kg.

3.4.2 | Precision and accuracy

Precision was examined by analysis of a spiked sample at three different concentrations (high, medium, and low) on the different days. The relative standard deviation values were from 2.3 to 7.6% (see Table 3). Method accuracy was evaluated by recovery studies using the blank sample free from triadimenol. Four kinds of triadimenol stereoisomers standard in different concentrations (high, medium, and low) were added into the blank samples (see Figure 3a and b). Results showed that the recovery rate of the four kinds of triadimenol stereoisomers ranged between 81.0 and 107% (see Table 3). According to SANTE/12682/2019 [33], the limit of quantitation was defined as lowest concentration level validated with satisfactory recoveries between 80 and 120% with an acceptable RSD in method validation part.

3.5 | Real samples

To observe the effectiveness and practicability of this method, the established method was applied to test the four kinds of triadimenol stereoisomers in ten randomly selected three pumpkin purees, four apple purees, and three tomato purees samples, which were purchased from local supermarkets. Results showed that four kinds of triadimenol stereoisomers were detected in an apple

TABLE 2 Linear equation, correlation coefficient, and LOQ of each compound

Compound	Linear range/(mg/L)	Linear equation	Correlation coefficient	LOQ (mg/kg)
(+)-(*R*,*S*)-Triadimenol	1.0 - 50	$y = 7.03 \times 10^3 x + 1.32 \times 10^3$	0.9997	0.05
(-)-(*S*,*R*)-Triadimenol	1.0 - 50	$y = -7.67 \times 10^3 x + 1.72 \times 10^3$	0.9997	0.05
(-)-(*S*,*S*)-Triadimenol	1.0 - 50	$y = 8.17 \times 10^3 x + 1.21 \times 10^3$	0.9998	0.05
(+)-(*R*,*R*)-Triadimenol	1.0 - 50	$y = -8.19 \times 10^3 x + 1.93 \times 10^3$	0.9997	0.05

TABLE 3 Spiked recoveries and RSDs of four kinds of triadimenol stereoisomers in pumpkin puree, apple puree, and tomato puree matrixes ($n = 6$)

		Pumpkin puree		Apple puree		Tomato puree	
Triadimenol enantiomer	Spiked/ (mg/kg)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
(+)-(*R*, *S*)-Triadimenol	0.05	82.6–97.2	6.8	85.2–102	6.2	84.6–103	6.5
	0.2	84.7–98.9	6.4	81.0–98.5	5.8	86.4–102	4.8
	1.0	84.4–94.6	2.8	83.6–99.2	4.4	81.6–95.4	2.3
(-)-(*S*, *R*)-Triadimenol	0.05	83.2–102	6.1	83.4–98.7	7.5	83.8–107	7.2
	0.2	85.2–90.1	4.0	87.0–101	7.0	84.0–105	5.4
	1.0	86.2–95.8	3.6	85.2–97.5	4.1	82.7–98.4	3.6
(-)-(*S*,*S*)-Triadimenol	0.05	82.1–102	5.9	83.7–101	6.9	83.0–96.8	6.8
	0.2	81.8–97.5	6.0	81.4–104	7.6	86.8–102	6.6
	1.0	86.4–103	4.0	85.4–98.7	3.9	84.2–99.8	4.5
(+)-(*R*,*R*)-Triadimenol	0.05	83.6–95.6	7.1	86.7–105	6.8	84.6–99.0	6.9
	0.2	81.5–97.3	6.6	81.8–103	6.5	83.8–106	7.6
	1.0	87.8–98.1	3.5	84.6–101	5.4	82.6–93.3	4.9

puree sample, in which, the content of (+)-(*R*,*S*)- triadimenol was 1.0 mg/kg, the content of (-)-(*S*,*R*)- triadimenol was 1.0 mg/kg, the content of (-)-(*S*,*S*)-triadimenol was 0.23 mg/kg, and content of (+)-(*R*,*R*)- triadimenol was 0.21 mg/kg (Figure 3c). It complied with the ratio of the four kinds of triadimenol stereoisomers in triadimenol raceme reported in literature [34], namely triadimenol A((+)-(*R*,*S*)- triadimenol and (-)-(*S*,*R*)- triadimenol):triadimenol B ((-)-(*S*,*S*)- triadimenol and (+)-(*R*,*R*)- triadimenol) = 4:1.

4 | CONCLUDING REMARKS

This paper separated the four kinds of triadimenol stereoisomers using UPC^2, and tested the residue of the

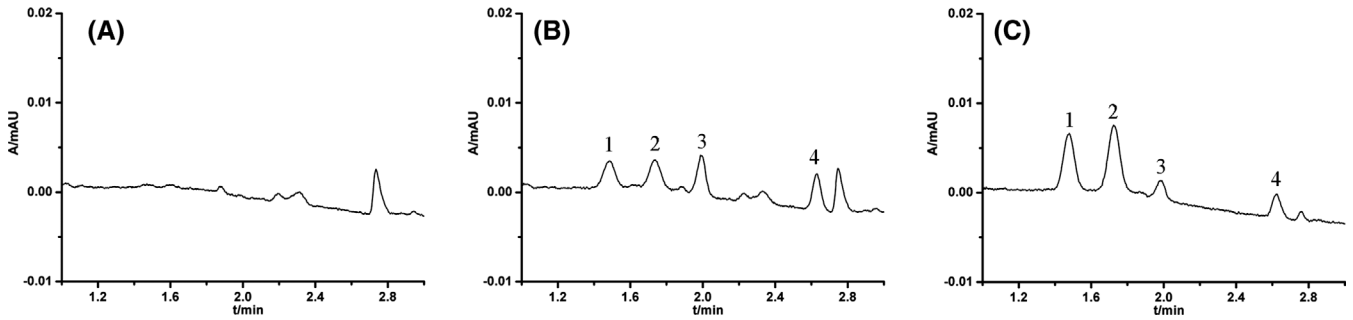

FIGURE 3 Chromatograms of (a) blank apple puree sample, (b) the sample spiked with 0.05 mg/kg four kinds of triadimenol stereoisomers standard, and (c) the actual positive apple puree sample. Peak 1: (+)-(*R*,*S*)-triadimenol; Peak 2: (-)-(*S*,*R*)-triadimenol; Peak 3: (-)-(*S*,*S*)-triadimenol; Peak 4: (+)-(*R*,*R*)-triadimenol

four kinds of triadimenol stereoisomer in fruit and vegetable puree represented by pumpkin puree, apple puree, and tomato puree. Acetonitrile was adopted for the extraction of samples, while graphitized carbon black and Florisil column were adopted for the purification of samples. Acquity Trefoil AMY1 was adopted for chiral chromatographic column separation, and gradient elute was carried out with supercritical carbon dioxide-methanol as the mobile phase and with external standard method for quantitation. Recovery experiment was carried out within the scope of 0.05–1.0 mg/kg, the standard recovery rate of the four kinds of triadimenol stereoisomers was 81.0–107%, and RSD was 2.3–7.6%. This method was applied to test the triadimenol stereoisomer residue in ten fruit and vegetable puree samples purchased from the market, and the detectable amount was from 0.21 to 1.0 mg/kg.

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CONFLICT OF INTEREST

The authors have declared no conflict of interest.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

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