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Residue and dissipation of epoxiconazole in *Triticum aestivum* L. and soil under field conditions

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Abstract: The dissipation dynamics of epoxiconazole in wheat plant and soil were evaluated, as well as the terminal residues of epoxiconazole in wheat grains, wheat plants and soil. A simple and effective analytical method for determining epoxiconazole residues in wheat grain, wheat plant and soil was established. The residue levels and dissipation rates of epoxiconazole in wheat and soil were determined by gas chromatography equipped with an electron capture detector (GC-ECD). At fortified levels of 0.01, 0.1 and 2 mg/kg in wheat grains and soil, and 0.01, 0.1 and 10 mg/kg in wheat plant, the recoveries ranged from 82% to 93%, with relative standard deviations (RSD) from 3.0% to 9.7%. The limits of quantification (LOQ) were 0.01 mg/kg for soil, wheat grains and wheat plants. The half-lives of epoxiconazole in wheat plant and in soil were 3.5-8.4 days and 10-30 days, respectively. When at the interval to harvest of 21 days，application rate of 112.5 g a.i./ha and two subsequent applications, the residue levels in wheat grain were below 0.05 mg/kg，and the terminal residues at harvest in wheat grain were lower than the maximum residue limits (0.05 mg/kg) for this pesticide set in China. When used in wheat, the following GAP was recommended: 21 days interval to harvest from last application, two maximum applications, with application rate of 112.5 g a.i./ha.

Keywords: epoxiconazole; wheat; soil; residues; dissipation dynamics

氟环唑在小麦及土壤中的残留及消解动态

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摘 要：为了评价氟环唑在小麦生产上使用的残留安全性，建立了气相色谱-电子捕获检测器检测氟环唑在小麦植株、小麦籽粒及土壤中残留的分析方法，并对氟环唑在小麦植株、小麦籽粒和土壤中的最终残留量及小麦植株和土壤中的消解动态进行了研究。结果表明：在添加水平为 0.01、0.1 和 2 mg/kg (小麦籽粒和土壤) 和 0.01、0.1 和 10 mg/kg (小麦植株) 下，氟环唑的回收

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率为 82%~93%，相对标准偏差为 3.0%~9.7%。氟环唑在小麦植株、小麦籽粒和土壤中的定量限均为 0.01 mg/kg。氟环唑在小麦植株和土壤中的消解半衰期分别为 3.5~8.4 和 10~30 d。当以有效成分 112.5 g/hm^2 的剂量施药 2 次、采收间隔期为 21 d 时，小麦籽粒中氟环唑的残留量为 <0.05 mg/kg，低于中国制定的小麦中氟环唑的最大残留限量值(0.05 mg/kg)。建议氟环唑在小麦上使用时最大剂量为有效成分 112.5 g/hm^2，施药 2 次，安全间隔期为 21 d。

关键词: 氟环唑；小麦；土壤；残留；消解动态

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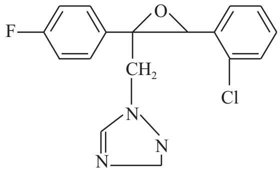
Wheat, which is cultivated throughout China, ranks as the third leading crop in the country following rice (*Oryza sativa*) and maize (*Zea mays*)[1]. There are many diseases in wheat, and the yield loss caused by those diseases are serious. Currently, fungicides have played a prominent role in cereal disease control.

Epoxiconazole (**Scheme 1**) is a highly efficient systemic triazole fungicide developed by BASF (Germany) in 1993[2-3]. It is used as a broad-spectrum fungicide with preventive and curative action and is extensively used worldwide to control diseases caused by *Ascomycetes*, *Basidiomycetes*, and *Deuteromycetes* in cereals, rice, bananas, coffee, soybean, sugar beets, peanut, oilseed rape, apple, and ornamentals[4]. It is also used to regulate plant growth by acting as an inhibitor of C-14-demethylase in sterol biosynthesis. Due to the extensive use of this fungicide on crops, there is a potential risk in coastal ecosystems located close to agricultural areas and the entrance into food chains for humans and wildlife[5-6].



Scheme 1

To the best of our knowledge, the residue analysis of epoxiconazole has been reported in various matrix such as rice plant and polished rice[7-9], vegetables and mosquito fish[7], soil[7-16], water[7-8, 12, 16], banana[14], wheat[15, 17], tubifex[16], wine[18], apple[19], fruit[20] and tea[20-21], grape[22], spicy flavor vegetables[23].

Lin et al.[7] established the method for the determination of epoxiconazole residues in field water, rice plant, polished rice, vegetables, soil, and mosquito fish using gas liquid chromatography with an electron-capture detector. The assay employs processing of field water extracted by *n*-hexane, rice plant or vegetables extracted by aqueous methanol solution, and cleaned up by silica gel column. Rybár et al.[13] developed an analytical method for the fast trace microanalysis of the chiral pesticides epoxiconazole in soil samples using reversed-phase high performance liquid chromatography. Fan et al.[15] developed a method to determine the residues in wheat and soil based on ultra-performance liquid chromatography-electrospray ionization tandem mass spectrometry (UPLC-MS/MS). Liu et al.[16] developed a method to determine residues in tubifex and soil based on high-performance liquid chromatography coupled with triple-quadrupole mass spectrometry (HPLC-MS/MS).

The present study developed a method to determine of epoxiconazole based on gas chromatography using an electron capture detector (GC-ECD). The price of GC-ECD is cheap, which is also widely used in analytical labs. Although the epoxiconazole dissipation have been studied in different matrices, such as wheat plants[15], grape and soil[24], and banana[14], no research regarding the epoxiconazole residues in wheat grains, wheat plants and soil in China is reported. Based on the available literature, the dissipation dynamics and the behavior of terminal residues of epoxiconazole in wheat and soil under field conditions were investigated. In China, the MRL value in wheat is 0.05 mg/kg. The purpose of this study was to determine whether epoxiconazole usage is safe under the recommended application methods. These results will aid the government in providing guidance concerning the proper and safe use of epoxiconazole.

1 Materials and methods

1.1 Materials

Epoxiconazole standard (99.2% purity) was purchased from the Sigma-Aldrich Corporation, USA. An epoxiconazole 125 g/L suspension concentrate (SC) was provided by Jiangsu Qizhoulu Chemical Co., Ltd., Jiangsu, China.

Acetonitrile and *n*-hexane (HPLC grade) for chromatography were purchased from Thermo Fisher Co., Ltd., USA. Analytical-grade acetone was purchased from Luoyang Chemical Reagent Factory, Henan, China. Analytical-grade sodium chloride was purchased from Beijing Reagent Company, Beijing, China.

The wheat variety in this investigation was Zhengmai 9023.

1.2 Experimental design

The field trails were conducted in a randomized block design in farmers' fields, which were previously investigated to be free of the pesticide. The supervised field trials were carried out in Henan Province and Zhejiang Province and in Beijing City, China, from 2009 to 2010. The field treatments included the dissipation experiment and final residue experiment. Each treatment consisted of 1 control and 3 replicates of treated samples with an area of 30 m^2. 2 metres wide buffer area was used to separate different treatments in the field. None of the plots had been treated with epoxiconazole in the past. To ensure the reliability of the experimental results, field management was carried out in accordance with local methods. During the trial, the daily average temperatures in Henan, Zhejiang and Beijing in the year of 2009 were 18.5, 19.25 and 15.25 ℃, respectively; the daily average temperatures in Henan, Zhejiang and Beijing in the year of 2010 were 17, 19.5 and 17.5 ℃, respectively. The rainfalls in Henan, Zhejiang and Beijing in the year of 2009 were 118.37, 479.32 and 96.74 mm, respectively; the rainfalls in Henan, Zhejiang and Beijing in the year of 2010 were 84.07, 384.04 and 131.56 mm, respectively. Physicochemical characterizations of the three types of soils are presented in Table 1.

Table 1 The physicochemical characterization of the three types of soils in Henan, Zhejiang and Beijing, China

Origin of the soil	Henan	Zhejiang	Beijing
Longitude	113°39′E	120°12′E	116°28′E
Latitude	35°9′N	30°16′E	39°58′N
Soil texture	Alluvial soil	Clay	Alluvial soil
pH (suspended 2:5 in water)	7.0	6.8	7.7
Total organic C content/%	1.2	1.8	1.4

To investigate the dissipation of epoxiconazole in wheat plants and in soil, when the wheat plant was at beginning of tillering, epoxiconazole (SC, 125 g/L) was dissolved in water and sprayed onto the surfaces of wheat plants using a manual sprayer (PS16-7, 40.5 cm × 17 cm × 57 cm, 16 L volume, max. pressure 1.0 MPa) at a application rate of 225 g a.i./ha (2 times of the highest recommended application rate). A plot of the same size but with no epoxiconazole application was conducted simultaneously. For the soil treatments, soil with no plants was sprayed at a application rate of 1 000 g a.i./ha. At 2 h and 1, 3, 5, 7, 10, 14, 21, 28, 35, 42 or 45, and 60 days after the spraying, approximately 200 g of wheat plants was randomly collected from several points in each plot, and an approximately 1 000 g soil sample was randomly sampled (at a depth of 0-10 cm) in each plot using a soil sampling apparatus (soil-sampling drill, i.d. 25 mm × height 20 cm) at 20 different points. Control samples were obtained from the control plots.

To investigate the terminal residues of epoxiconazole in wheat plants, wheat grains and soil, when the wheat plant was at beginning of flowering, two application rates were used: a low application rate at 112.5 g a.i./ha (1 time of the highest recommended application rate) and a high application rate at 168.75 g a.i./ha (1.5 times of the highest recommended application rate). Both the low and high application rates were used in two or three applications (7 day intervals each). The wheat plants, wheat grains and soil were sampled at 7, 14 and 21 days after the last spraying, and the final residues were analyzed. For the control treatment, samples without pesticide were collected.

Immediately after collecting, the samples were put into polyethylene bags and transported to the laboratory. The wheat grains were separated from the wheat spikes and concentrated using the cone-and-quartering method, after which, 200 g of the grain sample was ground into a coarse powder using a vegetation disintegrator. The soil samples were sifted through a 40-mesh sieve, and 100 g of the soil was concentrated using the cone-and-quartering method. The wheat plants were cut into small pieces (approximately 0.5 cm × 0.5 cm) prior to analysis. All samples were stored at –20 ℃ until analyzed.

1.3 Analytical methods

Soil and wheat grains: 10 g subsample of homogeneous soil or wheat grains was put into a 50 mL centrifuge tube. 10 mL distilled water was added, followed by the addition of 20 mL acetonitrile. The centrifuge tube was then placed on an ultrasonic machine, and the sample was extracted for 30 min. After extraction, 3 g sodium chloride was added, and the sample was vortexed for 1 min at high speed on a vortexer. The mixture was centrifuged at 4 000 r/min for 5 min. The supernatant (20 mL of the acetonitrile layer) was transferred to a 50 mL glass beaker and then concentrated to less than 1 mL on a rotary evaporator at 40 ℃. The supernatant was further evaporated under a stream of nitrogen until dry. The residues were redissolved in 2 mL *n*-hexane for clean-up.

Wheat plants: 10 g subsample of homogeneous wheat plants was placed into a 250 mL glass bottle with a lid, and 10 mL of distilled water was added, followed by the addition of 40 mL acetonitrile. The glass bottle was placed on an ultrasonic machine, and the sample was extracted for 30 min. The extraction was then filtered into a mixing cylinder with a stopper along with 3 g sodium chloride, shaken for 1 min, and incubated for 1 h. The supernatant (20 mL of the acetonitrile layer) was transferred to a 100 mL glass beaker. The above method was identical to those described for the soil and wheat grain samples.

Clean-up was performed using a pre-activated Florisil® cartridge eluted with 5 mL acetone/*n*-hexane (2:8, *V*/*V*) and 5 mL *n*-hexane. The epoxiconazole residues were adsorbed onto the Florisil® cartridge and then eluted off the cartridge with 5 mL of acetone/*n*-hexane (2:8, *V*/*V*). The solvent in the eluate was evaporated to dryness in a nitrogen evaporator with a water bath at (50±1) ℃. The residues were redissolved in 2 mL of *n*-hexane and transferred into a 2 mL glass autosampler vial for GC-ECD analysis.

The samples were analyzed using a GC (Thermo Trace GC, America) equipped with an electron capture detector (ECD). The column used for epoxiconazole determination was a DB-608 column (0.25 mm film thickness, 30 m length, 0.32 mm i.d., Thermo Scientific, USA). The instrument conditions were as follows: the injector temperature was held at 230 ℃, the oven temperature was initially 180 ℃ for 1 min, then increased to 220 ℃ at a rate of 30 ℃/min and maintained for 1 min, finally increased to 270 ℃ at a rate of 5 ℃/min and maintained for 5 min. Ultra-pure nitrogen was used as the carrier gas, and injections were performed in splitless mode.

1.4 Statistical analysis

The dissipation of epoxiconazole was described using a first-order kinetic equation, $c_t = c_0 \times e^{-kt}$. The dissipation half-lives ($t_{1/2}$) of epoxiconazole in each experiment was obtained using the function $t_{1/2} = \ln2/k$, where c_t is the concentration (mg/kg) at time t (days) after application, c_0 is the initial concentration (mg/kg), and k is the first-order rate constant (days^{-1}).

2 Results

2.1 Method validation

A standard calibration curve of epoxiconazole was constructed by plotting the analyzed concentrations against the peak areas. Good linearity was achieved in the range of 0.01 to 10 mg/L, standard curve was $y = 2.441 \times 10^6 x - 1.736 \times 10^4$, with a correlation coefficient of $R^2 = 0.999\ 4$. The recovery study was conducted for different matrix at three different fortified levels (0.01, 0.1, 2 or 10 mg/kg).

The average recoveries from fortified samples in five replicate experiments for each matrix ranged from 82% to 93%, with a relative standard deviation (RSD) from 3.0% to 9.7%. The limits of quantification (LOQ) were 0.01 mg/kg for all samples. The LOQ and recoveries obtained can meet Chinese national standards[25]. This result suggests that epoxiconazole could be detected with good precision and had good recoveries using the extraction procedure adopted.

2.2 Dissipation of epoxiconazole in wheat plants and soil

The half-lives and other statistical parameters of the epoxiconazole residue dissipation were calculated from the experimental data and were summarized in Table 2.

Table 2 First-order kinetic equations, half-lives and other statistical parameters for epoxiconazole dissipation under field conditions

Locality	Year	Matrix	Regression equation	Determination coefficient, R^2	Initial concentration/(mg/kg)	Half-lives/d
Henan	2009	Wheat plant	$1.456e^{-0.160\,7t}$	0.87	2.1	4.3
		Soil	$1.201e^{-0.040\,8t}$	0.96	0.99	17
	2010	Wheat plant	$2.607e^{-0.082\,2t}$	0.90	2.8	8.4
		Soil	$1.637\,9e^{-0.034\,4t}$	0.98	1.0	20
Zhejiang	2009	Wheat plant	$2.046e^{-0.128\,1t}$	0.96	2.6	5.4
		Soil	$2.738e^{-0.065\,7t}$	0.94	2.2	10
	2010	Wheat plant	$1.247e^{-0.195\,5t}$	0.95	2.1	3.5
		Soil	$4.307e^{-0.041\,1t}$	0.96	2.5	17
Beijing	2009	Wheat plant	$1.838e^{-0.126t}$	0.73	7.8	5.5
		Soil	$0.884\,2e^{-0.023\,4t}$	0.84	0.87	30
	2010	Wheat plant	$2.703e^{-0.156\,8t}$	0.80	9.2	4.4
		Soil	$0.762\,0e^{-0.035\,6t}$	0.86	1.5	20

2.3 Terminal residue of epoxiconazole in wheat grains, wheat plants and soil

The residues of epoxiconazole in wheat grains, wheat plants and soil under different application rates, different frequencies, and different intervals to harvest are presented in Table 3. The residue levels in wheat grains were < 0.01-1.0 mg/kg at the 7th day, < 0.01-0.54 mg/kg at the 14th day, and < 0.01-0.075 mg/kg at the 21th day. The residue levels in wheat plants were < 0.01-1.5 mg/kg at the 7th day after spraying, < 0.01-1.1 mg/kg at the 14th day, and < 0.01-0.85 mg/kg at the 21th days. The residue levels in soil were < 0.01-0.62 mg/kg at the 7th day, 0.018-0.46 mg/kg at the 14th day, and < 0.01-0.42 mg/kg at the 21th day.

3 Discussion

3.1 Dissipation of epoxiconazole in plant and soil

The initial concentrations of epoxiconazole in wheat plants in Henan Province, Zhejiang Province and Beijing City was 2.1, 2.8, 2.6 mg/kg in the year of 2009, respectively and 2.1, 7.8, 9.2 mg/kg in the year of 2010, respectively. Based on the dissipation data obtained in 2009 and 2010 from the above-mentioned three sites, the different initial concentrations of epoxiconazole in wheat plants could attribute to the different plant sizes. The half-lives of epoxiconazole in wheat plants in Henan Province, Zhejiang Province and Beijing City in the year of 2009 and 2010 was 4.3-8.4, 3.5-5.4, and 4.4-5.5 d, respectively. Differences in the dissipation rate and half-lives of epoxiconazole in the wheat plants might caused by the weather conditions (such as temperature and rainfall) after the chemical application. Higher temperature and solar irradiance enhanced the dissipation rates and resulted in shorter half-lives of epoxiconazole in the crop[7]. Choi et al.[26] reported that the residue levels of fungicides rapidly decrease during the early stages of precipitation. After the early stages, the rate of decrease in the residue levels slowly. Other papers also reported the half-lives of epoxiconazole in banana and in soil were 7.2-9.9 and 8.0-10 d[14], respectively, and those in wheat plants

Table 3 Terminal residues of epoxiconazole in wheat grains, wheat plants and soil under field conditions in Henan, Zhejiang and Beijing, China in 2009 and 2010

Year	Days after spraying	Application rate/(g a.i./ha)	Number of times sprayed	Residue ± SD (mg/kg) (n = 3)								
				Henan			Zhejiang			Beijing		
				Wheat grains	Soil	Wheat plants	Wheat grains	Soil	Wheat plants	Wheat grains	Soil	Wheat plants
2009	7	112.50	2	0.035 ± 0.018	0.150 ± 0.056	0.031 ± 0.002	0.220 ± 0.039	0.370 ± 0.027	0.073 ± 0.011	0.087 ± 0.008	0.060 ± 0.006	1.000 ± 0.270
			3	0.046 ± 0.001	0.150 ± 0.044	0.048 ± 0.004	0.290 ± 0.014	0.400 ± 0.038	0.200 ± 0.019	0.045 ± 0.035	0.130 ± 0.036	1.100 ± 0.770
		168.75	2	0.048 ± 0.028	0.150 ± 0.062	0.190 ± 0.160	0.420 ± 0.110	0.430 ± 0.031	0.380 ± 0.110	0.031 ± 0.004	0.160 ± 0.059	1.400 ± 0.440
			3	0.047 ± 0.024	0.140 ± 0.069	0.450 ± 0.032	0.430 ± 0.100	0.620 ± 0.023	0.640 ± 0.260	0.051 ± 0.017	0.310 ± 0.100	1.500 ± 1.100
	14	112.50	2	0.040 ± 0.016	0.061 ± 0.029	0.017 ± 0.008	0.065 ± 0.007	0.210 ± 0.012	0.080 ± 0.008	0.020 ± 0.005	0.021 ± 0.009	0.730 ± 0.570
			3	0.035 ± 0.012	0.120 ± 0.008	0.087 ± 0.009	0.069 ± 0.048	0.310 ± 0.027	0.059 ± 0.024	0.037 ± 0.003	0.021 ± 0.010	0.990 ± 0.018
		168.75	2	0.027 ± 0.027	0.190 ± 0.062	0.210 ± 0.019	0.130 ± 0.044	0.400 ± 0.061	0.280 ± 0.009	0.032 ± 0.008	0.030 ± 0.014	1.000 ± 0.150
			3	0.033 ± 0.007	0.120 ± 0.051	0.440 ± 0.140	0.180 ± 0.044	0.460 ± 0.004	0.380 ± 0.046	0.037 ± 0.008	0.120 ± 0.062	1.100 ± 0.820
	21	112.50	2	0.021 ± 0.008	0.067 ± 0.021	0.030 ± 0.002	0.011 ± 0.002	0.068 ± 0.024	0.250 ± 0.330	0.032 ± 0.010	0.022 ± 0.005	0.140 ± 0.031
			3	0.025 ± 0.005	0.041 ± 0.027	0.084 ± 0.080	0.027 ± 0.010	0.260 ± 0.006	0.044 ± 0.008	0.053 ± 0.020	0.030 ± 0.015	0.190 ± 0.160
		168.75	2	0.038 ± 0.016	0.072 ± 0.022	0.110 ± 0.094	0.039 ± 0.032	0.380 ± 0.013	0.210 ± 0.004	0.051 ± 0.014	0.052 ± 0.014	0.300 ± 0.280
			3	0.032 ± 0.006	0.082 ± 0.033	0.190 ± 0.062	0.055 ± 0.026	0.420 ± 0.007	0.330 ± 0.045	0.027 ± 0.003	0.110 ± 0.049	0.850 ± 0.016
2010	7	112.50	2	0.430 ± 0.020	0.086 ± 0.000	0.017 ± 0.001	0.052 ± 0.009	0.130 ± 0.015	0.031 ± 0.015	<0.010	0.014 ± 0.007	0.040 ± 0.007
			3	0.700 ± 0.071	0.058 ± 0.006	<0.010 [a]	0.070 ± 0.021	0.180 ± 0.080	0.069 ± 0.045	<0.010	0.013 ± 0.005	0.040 ± 0.011
		168.75	2	1.000 ± 0.019	0.059 ± 0.014	<0.010	0.059 ± 0.022	0.310 ± 0.400	0.180 ± 0.047	0.026 ± 0.014	<0.010	0.150 ± 0.061
			3	1.000 ± 0.270	0.180 ± 0.007	<0.010	0.160 ± 0.067	0.290 ± 0.062	0.100 ± 0.051	0.035 ± 0.025	0.110 ± 0.032	0.410 ± 0.380
	14	112.50	2	0.059 ± 0.066	0.040 ± 0.023	0.014 ± 0.007	0.028 ± 0.004	0.056 ± 0.001	0.200 ± 0.063	<0.010	0.048 ± 0.010	0.042 ± 0.007
			3	0.300 ± 0.026	0.049 ± 0.004	0.016 ± 0.001	0.059 ± 0.020	0.063 ± 0.003	0.066 ± 0.008	<0.010	0.083 ± 0.014	0.012 ± 0.000
		168.75	2	0.340 ± 0.320	0.018 ± 0.008	<0.010	0.089 ± 0.014	0.160 ± 0.015	0.540 ± 0.200	<0.010	0.052 ± 0.005	0.170 ± 0.092
			3	0.540 ± 0.096	0.029 ± 0.018	<0.010	0.086 ± 0.065	0.150 ± 0.030	0.012 ± 0.001	0.011 ± 0.001	0.120 ± 0.015	0.180 ± 0.020
	21	112.50	2	0.013 ± 0.004	0.014 ± 0.003	0.016 ± 0.007	0.043 ± 0.009	0.084 ± 0.011	0.049 ± 0.007	0.026 ± 0.005	0.057 ± 0.013	0.016 ± 0.002
			3	0.062 ± 0.028	0.020 ± 0.002	0.014 ± 0.003	0.056 ± 0.063	0.086 ± 0.041	0.023 ± 0.008	<0.010	0.048 ± 0.019	0.016 ± 0.002
		168.75	2	0.031 ± 0.014	0.094 ± 0.037	<0.010	0.032 ± 0.002	0.260 ± 0.033	0.044 ± 0.035	0.023 ± 0.007	0.044 ± 0.034	0.011 ± 0.001
			3	0.056 ± 0.046	0.110 ± 0.018	0.058 ± 0.045	0.075 ± 0.004	0.350 ± 0.073	0.015 ± 0.005	0.028 ± 0.002	<0.010	0.120 ± 0.027

[a] < The limit of quantification (LOQ).

and in soil were 8.1-12.6 and 16.9-21.3 d[15] respectively. Half-lives of epoxiconazole ranged from 2.5 to 6.0 months under aerobic conditions[27].The field measurements of behavior were desirable, as simulations based on laboratory measurements can overestimate persistence[10-11]. The half-lives of epoxiconazole in soil in Henan Province, Zhejiang Province and Beijing City in the year of 2009 and 2010 was 17-20, 10-17, and 20-30 d, respectively. Differences in the dissipation rate and half-lives of epoxiconazole in the soil might due to local soil characteristics, farming methods, microbial activities and weather conditions. The epoxiconazole half-lives estimated in sandy loam soil tend to be shorter than those in clay loam soil[2], and the half-lives are shorter in alkaline soils than those in acidic soils. This study demonstrated that the dissipation of epoxiconazole in wheat plants was faster than that in soil. A growth dilution factor might play an important role in the pesticide dissipation in the plant, in addition to the effect of physical and chemical factors such as light, heat, pH value and moisture[28]. The growth dilution factor is important in reducing epoxiconazole residue levels in wheat fields because the residue is expressed as a proportion of weight (mg/kg). The wheat plants were picked in different stages of growth in this study, and as the weight of wheat plant material increases, the proportion of residue decreases[24].

3.2 Terminal residues of epoxiconazole

According to the terminal residue results, the residue behavior of epoxiconazole in grain, straw and soil under different treatments almost followed a trend in which higher epoxiconazole doses and shorter harvest intervals led to more residual epoxiconazole.

The terminal residues at harvest in the wheat grains, wheat plants and soil were lower than 1.0, 1.5 and 0.62 mg/kg, respectively. In this study, the order of residue levels was wheat grains < wheat plants, which was similar with that in Europe[29]. These results could be explained by the following reasons: 1) in the residue experiments, the epoxiconazole was sprayed directly on the wheat plants and not on the soil. Although the dissipation of epoxiconazole in wheat plants was faster than that in the soil, the moisture content in the wheat plant was also greatly reduced at harvest, causing the epoxiconazole residues per unit weight to be higher than that in soil and wheat. 2) Epoxiconazole is a systemic pesticide, so when it is sprayed onto the wheat plants, it can be transferred to wheat grains. Pesticide residues in the soil raise several potential risks, such as an adverse effect on subsequent crops and contamination of the groundwater[30]. In the final residue experiments, epoxiconazole residues in the soil were detectable at three locations, but the potential risks of these residues remain undetermined.

4 Conclusions

A specific, sensitive and simple residue analytical method using GC-ECD for the detection and monitoring of epoxiconazole in wheat grain, wheat plant and soil was established. The half-lives of epoxiconazole in soil and wheat plant were 3.5-8.4 and 10-30 d, respectively. When the interval to harvest was 21 d and epoxiconazole was sprayed at a low application rate and frequency, the terminal residues at harvest in the wheat grain were lower than 0.05 mg/kg.

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