



The dissipation rates of trichlorfon and its degradation product dichlorvos in cabbage and soil

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ABSTRACT

The residual levels and dissipation rate of trichlorfon, and its degradation product, dichlorvos, in cabbage crops and the soil in which these were grown, were determined by gas chromatography at two geographically distant experimental sites, one in Kunming and one in Beijing, China. Trichlorfon was applied at two dosages (900 g ai ha⁻¹ and 1350 g ai ha⁻¹). Maximum final residues of trichlorfon in soil and cabbage were 1.23 mg kg⁻¹ and 1.81 mg kg⁻¹ respectively at Kunming, and 0.35 mg kg⁻¹ and 0.70 mg kg⁻¹ respectively at Beijing. However, the final residues of dichlorvos in both cabbage and soil was only 0.04 mg kg⁻¹ at Kunming, and only 0.03 mg kg⁻¹, or “not detectable”, at Beijing. The mean half-life of trichlorfon in cabbage was 1.80 d with a dissipation rate of 90% over 5 d, while that in soil was 3.05 d with a dissipation rate of 90% over 14 d at one experimental site. The dissipation rates of trichlorfon and its degradation product dichlorvos at the two experimental sites were different, suggesting that degradation of these pesticides was affected by local soil characteristics and climate. When applied at both the recommended dosage and at 1.5 times this, no detectable residues of either trichlorfon or dichlorvos were found in soil or cabbage at harvest. Although trichlorfon can easily degrade into dichlorvos, which is highly toxic to humans and other animals, the observed low residual levels of dichlorvos suggest that trichlorfon is safe when applied at the recommended dosage.

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1. Introduction

Trichlorfon [o,o-dimethyl (2,2,2-trichloro-1-hydroxyethyl) phosphonate] is a broad-spectrum organophosphorus insecticide (Horsberg et al., 1990) used to control pest insects on numerous crops, including cabbage, which is an important vegetable in China and cultivated throughout the country. Trichlorfon can easily be degraded to the highly toxic insecticide dichlorvos (Barthel et al., 1955). Both trichlorfon and dichlorvos are highly polar organophosphorus pesticides (OPs), however, dichlorvos is about eight times more toxic than trichlorfon. The LD₅₀ of dichlorvos is 56–80 mg kg⁻¹ (rat, oral), while that of trichlorfon is 630 mg kg⁻¹. The physical properties of these two OPs are different; dichlorvos evaporates faster than trichlorfon at the same temperature (Dunier et al., 1991; Yamano and Morita, 1992).

Although there has been some research on trichlorfon residues in some animal and plant products (Ngoh and Cullison, 1996; Blasco et al., 2002; Brito et al., 2002; Grimalt et al., 2006), little work has been done on cabbage specifically, especially synchro-

nous monitoring of the degradation rates of trichlorfon and its degradation product. In this study, gas chromatography was used to synchronously monitor levels of trichlorfon, and its degradation product dichlorvos, in both cabbages and the soil these were grown in. The dissipation and kinetics of trichlorfon and residual levels of dichlorvos were determined to provide a reference for setting the maximum safe application rate of trichlorfon for cabbage crops in China.

2. Materials and methods

2.1. Materials

2.1.1. Pesticide

Trichlorfon emulsifiable (EC, 30%) was the pesticide used.

2.1.2. Crop

Cabbage was the vegetable used.

2.1.3. Chemicals

Acetone (99.5%), dichloromethane (99.5%), n-hexane (95.0%), toluene (99.5%), ethyl acetate (99.5%), sodium chloride (99.5%), and anhydrous sodium sulfate (99%) were all AR grade and

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purchased from Beijing Chemical Reagents Co. Ltd. (Beijing, China). Silica gel (60–100 mesh) obtained from Haiyang Chemical Factory (Qingdao, China) was baked for 4 h at 135 °C and deactivated with 1.5% distilled water before use. Standard trichlorfon (99.4% purity) and dichlorvos (99.0% purity) was obtained from China National Quality Control and Inspection Center for Pesticides (Beijing, China).

2.2. Experimental design

Field experiments were conducted in Beijing Municipality (39°–54′ north latitudes and 116°–28′ east longitudes) and Kunming in Yunnan province (25°–03′ north latitudes and 102°–42′ east longitudes), China in 2006 and 2007. Seven 15 m² trial plots (four residual plots, two dissipation plots and one control plot) were prepared at each site. Each plot type was replicated in triplicate making a total of 21 plots (12 residual, six dissipation and three controls) at each site. No trichlorfon was applied to the control plots.

2.3. Trichlorfon dissipation

To investigate the dissipation of trichlorfon in cabbage and soil, the trial plots were sprayed with trichlorfon, then sampled and tested for residues at different times. During the last 10 d of April in 2006 and 2007, trichlorfon (EC, 30%) was dissolved in water and sprayed onto the surface of cabbage and soil at a dosage of 1350 g ai ha⁻¹. At 0.042 (1 h after application), 0.33 (8 h after application), 1, 2, 3, 5, 7, 14, 21, and 30 d after spraying, 2 kg of cabbage was collected from 5 to 6 randomly selected sampling points within three replicate plots. Edible parts of the cabbage samples were cut into four and one selected at random for analysis. The sampling dates for the soil were the same as for the cabbage. Soil was sampled to a depth of 0–10 cm at 15 randomly selected sampling points in each plot using a soil-sampling auger. Both plant and soil samples were stored at –20 °C until analyzed.

2.4. Determination of final trichlorfon residues

The growth period of the cabbage used in this experiment, which was harvested in early summer, was about 2 or 3 months. To investigate the effect of trichlorfon dosage, spray frequency, and the residual level of trichlorfon in the environment, a two-by-two experimental design was used. High (1350 g ai ha⁻¹) and low (900 g ai ha⁻¹) dosage treatment plots had different spray frequencies; low dosage treatment plots were sprayed twice and high dosage treatment plots three times, with an interval of 7 d between each spraying. Soil and cabbage were sampled to determine the final residual level of trichlorfon and its degradation product dichlorvos. The soil in which the cabbage had been grown was sampled on day 3, 7, and 14 postspraying, respectively.

2.5. Analytical methods

2.5.1. Sample preparation

Twenty grams of cabbage or soil samples were tested for trichlorfon and dichlorvos residues using gas chromatography (GC) with a flame photometric detector (FPD). Cabbage samples were cut up and pulverized in a blender. The soil samples were measured on a dry matter basis after having been dried in the shade and sifted through a 40-mesh sieve.

2.5.2. Extraction

An aliquot of homogenized cabbage sample (20 g) was placed in a conical flask with 30 mL of acetone. The samples were then equilibrated overnight. After surging for 1 h, samples were filtered with 80 mL acetone using an 80-mm glass filter and air pump. The fil-

trate was combined and transferred to a funnel with a little acetone after which 200 mL of 5% sodium chloride and 50 mL dichloromethane were added and mixed for 2 min. The dichloromethane phase was collected and then transferred to a funnel with anhydrous sodium sulfate. The water phase was extracted by surging twice with dichloromethane (30, 30 mL). The extracts were combined in a round-bottom flask and concentrated to near dryness on a rotary vacuum evaporator (Model NE-1, EYELA Co., Tokyo, Japan) at 30 °C.

Twenty gram soil samples were mixed with 50 mL ethyl acetate and 25 g anhydrous sodium sulfate and then left overnight. After surging for 1 h, samples were filtered and the residues washed with 70 mL ethyl acetate. The organic layer from each sample was collected and then evaporated to near dryness on a rotary vacuum evaporator at 30 °C. The deposit (soil extract) was dissolved in 10 mL ethyl acetate for GC analysis.

2.5.3. Clean-up

The clean-up process for cabbage samples followed the modified Method S 19 (Specht et al., 1995). A small quartz wool plug was put in the bottom of the glass clean-up column (0.8 cm i.d.) to which a 4-cm-thick layer of anhydrous sodium sulfate, 1 g silica gel, and another 2-cm-thick layer of anhydrous sodium sulfate were successively added. The column was then rinsed with 5 mL n-hexane and the eluant discarded. The column was then eluted with solvents, or a mixture solvents, of increasing polarity.

One milliliter of n-hexane/toluene mixture (65:35, v/v) (HT solution) was transferred into a round-bottomed flask, and carefully placed in an ultrasonic bath to dissolve the residue on the glass surface. The resultant solution was then transferred to the clean-up column. This step was repeated twice and aggregate solution eluted with a further 5 mL of HT solution. This solution was then discarded. Another 1 mL of toluene was then transferred into a round-bottomed flask. The column was eluted as above with 10 mL of toluene and the resultant solution discarded. Another round-bottomed flask was placed under the column to which 6 mL of toluene/acetone mixture (95:5, v/v) was added and the column eluted. This step was repeated with a 10 mL of toluene/acetone mixture (80:20, v/v) and the resultant solutions were collected and concentrated to near dryness at 25 °C and made up to a final volume (10 mL) to assay for dichlorvos. Another volumetric flask containing acetone was placed under the column and the column eluted as above with acetone to a final volume (10 mL) to assay for trichlorfon.

2.5.4. GC analysis

Estimation of trichlorfon residues was performed using a GC (Model HP-5890 II) equipped with an HP-1 column (100% polydimethylsiloxane; 5 m × 0.53 mm i.d. × 0.25 μm film thickness). Other GC parameters were as follows: oven program 60 °C for 2 min, rising at 10 °C min⁻¹ to 150 °C, holding for 2 min; injection port 150 °C; detector (FPD) 250 °C; gas flow (mL min⁻¹), nitrogen 13, hydrogen 85, air 75. The relative retention times for trichlorfon and dichlorvos were 8.3 min and 5.7 min, respectively (Fig. 1).

2.5.5. Calculations

Different amounts of standard solution of trichlorfon (purity: 99.4%) and dichlorvos (purity: 98%) were added to cabbage and soil samples from the unsprayed control plots and the trichlorfon and dichlorvos residue in these samples determined by the external standard method. Recovery was determined with single-point calibration. There was a positive linear relationship ($y = 188\ 620X - 4531.6$, $R^2 = 0.9991$) between the peak area (y) for trichlorfon and its concentration (X) in the range of 0.03–2.00 mg L⁻¹ and a positive linear relationship ($y = 303\ 151X + 1329.8$, $R^2 = 0.9997$) for dichlorvos in the range of 0.006–0.400 mg L⁻¹.

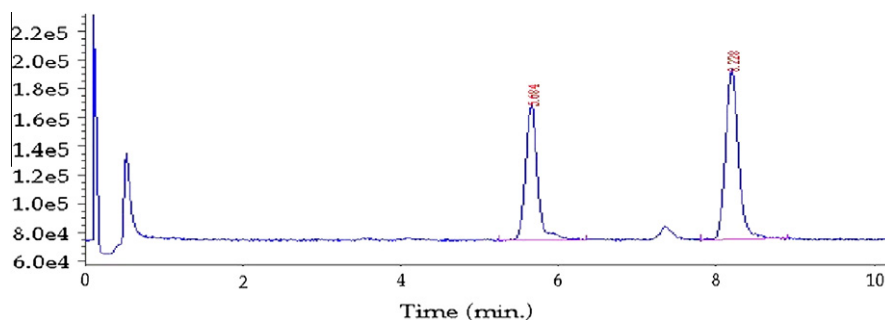


Fig. 1. Typical GC chromatograph of a standard solution of trichlorfon (1.5 mg L^{-1}) and dichlorvos (0.3 mg L^{-1}). The relative retention times for trichlorfon and dichlorvos were 8.3 min and 5.7 min, respectively.

The residual amount and half-life of trichlorfon were calculated by the equations; $C_T = C_0 e^{-KT}$ and $T_{1/2} = -\ln(0.5)/K$, respectively, in which T is the time (d or h) after application of the pesticide, C_T is the residue of the pesticide at time T , C_0 is an initial residue after application (at $T = 0$), K is a dissipation coefficient, and $T_{1/2}$ is the time required for the pesticide residue level to fall to half the initial level after application.

3. Results

3.1. Recovery

The recoveries of trichlorfon in soil and cabbage samples were 77–100% and 83–106%, respectively, and those of dichlorvos were 77–101% and 84–100%, respectively. Recovery data and their coefficients of variance were acceptable (Table 1). The limit of quantitation was defined as the minimum spiked level for trichlorfon or dichlorvos, and was 0.01 mg kg^{-1} for both substances.

Table 1
Recovery (%) of trichlorfon and dichlorvos from cabbage and soil from unsprayed control plots^a.

Sample type	Trichlorfon		Dichlorvos	
	Amount added (mg kg^{-1})	Recovery (%)	Amount added (mg kg^{-1})	Recovery (%)
Soil	0.01	96.40 ± 4.10	0.01	95.00 ± 3.94
	0.10	83.80 ± 5.40	0.10	93.80 ± 6.53
	1.00	84.00 ± 1.87	1.00	88.00 ± 7.68
Cabbage	0.01	89.20 ± 5.76	0.01	93.00 ± 4.69
	0.10	98.60 ± 4.98	0.10	92.00 ± 6.52
	1.00	94.40 ± 6.02	1.00	92.80 ± 2.77

^a Recovery (%) is the average of five repetitions.

Table 2
The dissipation of trichlorfon and its degradation product dichlorvos in cabbage and soil from experimental sites at Kunming and Beijing.

Sample sites	Samples	Pesticides	Dissipation	
			Degradation equations ($C_t = C_0 e^{-KT}$)	Half-life (d)
KM	Cabbage	TCF	$C = 9.14e^{-0.31T}$ ($r = 0.97$)	2.2
		DDVP	$C = 0.32e^{-0.59T}$ ($r = 0.95$)	1.2
	Soil	TCF	$C = 1.76e^{-0.19T}$ ($r = 0.98$)	3.7
		DDVP	$C = 0.06e^{-0.17T}$ ($r = 0.91$)	4.1
BJ	Cabbage	TCF	$C = 10.11e^{-0.46T}$ ($r = 0.99$)	1.4
		DDVP	$C = 0.13e^{-0.43T}$ ($r = 0.92$)	1.6
	Soil	TCF	$C = 0.59e^{-0.27T}$ ($r = 0.95$)	2.6
		DDVP	$C = 0.04e^{-0.46T}$ ($r = 0.96$)	1.5

Note: the numbers in parentheses are correlation coefficients.
Abbreviations: KM, Kunming; BJ, Beijing; TCF, trichlorfon; DDVP, dichlorvos.

3.2. Dissipation of trichlorfon in cabbage and soil

The degradation kinetics equations for trichlorfon and dichlorvos are shown in Table 2. The initial residue of trichlorfon in cabbage grown in Kunming was 9.3 mg kg^{-1} with a half-life ($T_{1/2}$) of 2.2 d; 95% of this initial residue had dissipated after 5 d. The initial residue of trichlorfon in the cabbage grown in Beijing was 10.3 mg kg^{-1} with a half-life of 1.4 d; 92% of this residue had dissipated after 5 d (Table 2 and Fig. 2). The initial residue of trichlorfon in soil from the Kunming experimental field was 1.8 mg kg^{-1} with a half-life of 3.6 d; 96% of this residue had dissipated after 14 d. However, the initial residues in soil from the Beijing field was 0.63 mg kg^{-1} with a half-life of 2.5 d; 94% of this residue had dissipated after 7 d (Table 2 and Fig. 2).

3.3. Final residues of trichlorfon and dichlorvos in cabbage and soil

Residual levels of trichlorfon in cabbage and soil in Kunming were 1.77 mg kg^{-1} and 1.27 mg kg^{-1} respectively and those in Beijing were 0.70 mg kg^{-1} and 0.35 mg kg^{-1} respectively (Tables 3 and 4). Trichlorfon residues in both cabbage and soil were much lower in Beijing than in Kunming. Most residual levels of dichlorvos in cabbage and soil in Kunming were below 0.01 mg kg^{-1} and 0.04 mg kg^{-1} respectively; similarly, most residual levels of trichlorfon in cabbage and soil in Beijing were less than 0.01 mg kg^{-1} (Tables 3 and 4). Levels of both trichlorfon and dichlorvos decreased faster in Beijing than Kunming.

4. Discussion

Initial residues of trichlorfon in cabbage and soil differed between the two experimental sites. The cabbage grown in Kunming had many times the residual level of trichlorfon as those grown in Beijing. The dissipation of trichlorfon in cabbage samples from Beijing was faster than that in Kunming (95% vs 92% 5 d after application). Similarly, dissipation of trichlorfon in soil was also faster in Beijing than in Kunming (2.5 d vs 3.6 d). This difference probably reflects the lower initial residual level of trichlorfon in the soil at Beijing compared to Kunming (0.63 mg kg^{-1} vs 1.80 mg kg^{-1}). These between-site differences suggest that local soil characteristics and climate may affect the dissipation of trichlorfon (Li et al., 2008). Usually, the degradation of trichlorfon in soil is affected by many factors, such as soil type, pH, and organic matter content (Chapman and Cole, 1982; Petrovic et al., 1993; Li et al., 2006). With regard to the degradation of trichlorfon in cabbage plants, in addition to physical and chemical factors, agrometeorological conditions, such as rainfall, moisture, and sunshine hours may have played a significant role. Annual sunshine hours (ASH) are an important factor affecting the photodegradation rate of pesticides in the environment and Beijing has more ASH (578 h) than

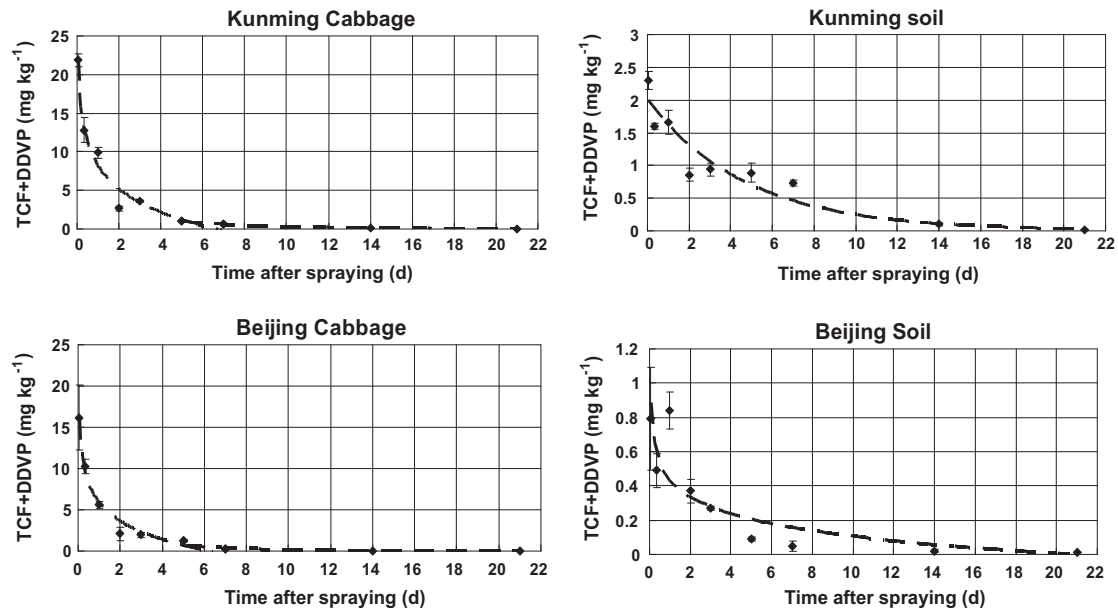


Fig. 2. Dissipation of trichlorfon residues in soil of experimental cabbage fields and cabbage grown in Kunming and Beijing, China. The residues of trichlorfon (TCF) in the samples tested include its degradation product, dichlorvos (DDVP), which is indicated as TCF + DDVP.

Table 3

Final residues of trichlorfon and its degradation product dichlorvos in cabbage grown in Kunming and Beijing, China.

Days after spraying	Number of times sprayed	Dosage (g ai ha ⁻¹)	Kunming				Beijing			
			2006		2007		2006		2007	
			TCF	DDVP	TCF	DDVP	TCF	DDVP	TCF	DDVP
3	2	900	0.66 ± 0.26	<0.01	0.68 ± 0.10	<0.01	0.55 ± 0.34	<0.01	0.13 ± 0.04	<0.01
		1350	1.60 ± 0.30	0.04 ± 0.01	1.12 ± 0.18	0.02 ± 0.00	0.60 ± 0.29	<0.01	0.09 ± 0.03	<0.01
	3	900	0.84 ± 0.19	<0.01	0.56 ± 0.11	<0.01	0.70 ± 0.28	<0.01	0.09 ± 0.02	<0.01
		1350	1.49 ± 0.27	0.03 ± 0.01	1.77 ± 0.31	0.04 ± 0.01	0.56 ± 0.01	<0.01	0.12 ± 0.00	<0.01
7	2	900	0.02 ± 0.01	<0.01	0.04 ± 0.01	<0.01	0.10 ± 0.02	<0.01	0.03 ± 0.00	<0.01
		1350	0.05 ± 0.01	<0.01	0.04 ± 0.01	<0.01	0.26 ± 0.07	<0.01	0.06 ± 0.01	<0.01
	3	900	0.03 ± 0.01	<0.01	0.03 ± 0.00	<0.01	0.08 ± 0.02	<0.01	0.04 ± 0.01	<0.01
		1350	0.05 ± 0.01	<0.01	0.05 ± 0.05	<0.01	0.13 ± 0.04	<0.01	0.05 ± 0.01	<0.01
14	2	900	0.01 ± 0.00	<0.01	0.01 ± 0.01	<0.01	0.02 ± 0.00	<0.01	0.03 ± 0.00	<0.01
		1350	<0.01	<0.01	0.01 ± 0.01	<0.01	0.04 ± 0.00	<0.01	0.03 ± 0.02	<0.01
	3	900	<0.01	<0.01	<0.01	<0.01	0.03 ± 0.00	<0.01	0.03 ± 0.00	<0.01
		1350	<0.01	<0.01	<0.01	<0.01	0.04 ± 0.01	<0.01	0.03 ± 0.00	<0.01

Abbreviations: TCF, trichlorfon; DDVP, dichlorvos.

Table 4

Final residues of trichlorfon and its degradation product dichlorvos in soils of experimental cabbage fields in Kunming and Beijing, China.

Days after spraying	Number of times sprayed	Dosage (g ai ha ⁻¹)	Kunming				Beijing			
			2006		2007		2006		2007	
			TCF	DDVP	TCF	DDVP	TCF	DDVP	TCF	DDVP
3	2	900	0.30 ± 0.05	0.01 ± 0.01	0.55 ± 0.24	0.01 ± 0.00	0.08 ± 0.06	<0.01	0.16 ± 0.02	0.01 ± 0.00
		1350	1.16 ± 0.43	0.03 ± 0.01	0.55 ± 0.06	0.01 ± 0.00	0.07 ± 0.02	<0.01	0.26 ± 0.07	0.03 ± 0.01
	3	900	0.70 ± 0.24	0.02 ± 0.00	0.59 ± 0.01	<0.01	0.10 ± 0.04	<0.01	0.12 ± 0.03	0.01 ± 0.00
		1350	1.27 ± 0.45	0.04 ± 0.00	1.23 ± 0.45	0.01 ± 0.00	0.08 ± 0.01	<0.01	0.35 ± 0.13	0.03 ± 0.01
7	2	900	0.10 ± 0.04	<0.01	0.46 ± 0.09	0.01 ± 0.00	0.02 ± 0.03	<0.01	0.07 ± 0.02	0.01 ± 0.00
		1350	0.28 ± 0.12	<0.01	0.48 ± 0.14	<0.01	0.03 ± 0.01	<0.01	0.15 ± 0.05	0.02 ± 0.00
	3	900	0.13 ± 0.04	<0.01	0.57 ± 0.23	0.01 ± 0.00	0.02 ± 0.00	<0.01	0.07 ± 0.03	0.01 ± 0.00
		1350	0.22 ± 0.02	<0.01	0.93 ± 0.45	0.02 ± 0.02	0.02 ± 0.00	<0.01	0.14 ± 0.02	0.01 ± 0.00
14	2	900	0.04 ± 0.00	<0.01	0.15 ± 0.04	<0.01	0.01 ± 0.00	<0.01	0.01 ± 0.00	<0.01
		1350	0.06 ± 0.01	<0.01	0.23 ± 0.04	<0.01	0.01 ± 0.00	<0.01	0.03 ± 0.01	<0.01
	3	900	0.05 ± 0.00	<0.01	0.27 ± 0.04	<0.01	0.01 ± 0.00	<0.01	0.02 ± 0.02	<0.01
		1350	0.09 ± 0.03	<0.01	0.22 ± 0.09	<0.01	0.02 ± 0.01	<0.01	0.04 ± 0.01	<0.01

Abbreviations: TCF, trichlorfon; DDVP, dichlorvos.

Kunming (476 h). This alone could explain the faster dissipation of trichlorfon in cabbage and soil in Beijing compared to Kunming.

Despite the fact that trichlorfon dissipated more slowly in soil than in cabbage, residues in soil were much lower than those in cabbage. Residual levels of dichlorvos, a much more toxic chemical, were very low in both cabbage and soil at both experimental sites. FAO/WHO has not established maximum residual limits (MRL) for trichlorfon, but in some countries, such as Germany and Japan, the MRLs of trichlorfon in fruits, vegetables, and cereals have been stipulated to be 0.05–0.10 mg kg⁻¹. The acceptable daily intake of trichlorfon is 0.01 mg kg⁻¹ body weight (Exttoxnet, 1998). Our results provide a quantitative basis for revising the guidelines for the application of this pesticide to vegetables. We recommend that cabbage be sprayed no more than twice with trichlorfon (EC, 30%) at a dosage not exceeding 900 g ai ha⁻¹ with an interval of at least 7 d between each application. There should be a withholding period of at least 14 d between the last application and harvest.

5. Conclusions

The dissipation rates of trichlorfon and its degradation product dichlorvos at the two experimental sites were different, suggesting that degradation of these pesticides was affected by local soil characteristics and climate. Although trichlorfon can easily degrade into dichlorvos, which is highly toxic to humans and other animals, the observed low residual levels of dichlorvos suggest that trichlorfon is safe when applied at the recommended dosage.

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