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Residue and persistence of insecticide 1-(2-chlorobenzoyl)-3-(4-chlorophenyl) urea in Chinese cabbage and soils in the Northern China

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Abstract — The residue and persistence of the insect growth regulator 1-(2-(chlorobenzoyl)-3-(4-chlorophenyl) urea (CCU) in Chinese cabbage and soil in the field were studied in Northern China during 1988–1989. The degradation of CCU in soils was also studied under laboratory conditions. The CCU was found to be unstable in Chinese cabbage and soil in field. The half-life of CCU was 13.2–14.0 days in Chinese cabbage and 8.8–27.0 days in soil, respectively. Three ppm as the maximum residue level for CCU in Chinese cabbage and 21 days as the preharvest interval for the vegetable treated with CCU were recommended.

Keywords: pesticide; 1-(2-chlorobenzoyl)-3-(4-chlorophenyl) urea; residue; Chinese cabbage; soil.

INTRODUCTION

The insect growth regulator 1-(2-chlorobenzoyl)-3-(4-chlorophenyl) urea (CCU) synthesized by Jiangsu Normal College, China in 1976 has been widely used to control the pine moth of forestry, armyworm of wheat and corn cabbage, caterpillar of vegetable and so on (Chen, 1980; 1981; 1984; Lu, 1981). Similar to insecticide diflubenzuron, UUC had novel and highly specific activity against a wide variety of insects. This kind of compound acted by interfering with the chitin metabolism of caterpillars and larvae, preventing the shedding of skins, leading to the death of the pupae or to non-viable adults, and preventing the eggs hatching (Royal Societyof Chemistry, 1983). CCU was a low toxic compound. Its acute oral LD_{50} for rats and mice was greater than 10000mg/kg. It did not effect on distortion and change, and was not toxic to birds, fishes and bees (Sun, 1982; Lu, 1983). As it was likely to have extensive usage, we reported here on the residue and persistence of CCU in Chinese cabbage and soil in the field in Northern China.

MATERIALS AND METHODS

Chemicals

Formulation of CCU used in the experiment was obtained from Tonghua Pesticide Plant, Jilin Province of China. The standard compound of CCU with purity of 99% was obtained by purifying of the formulation of CCU in our laboratory. Mathanol and dichloromethane used were analytical grade.

Degradation of CCU in soils under laboratory conditions

Standard compound of CCU was mixed with 10g of soil (10% moisture). The treated soil was incubated in aerobic condition at 28°C for 48 days. The concentration of CCU in soil was detected at regular intervals. The characteristics of soils used in the experiments are listed in Table 1.

Table 1 Characteristics of soil samples

Soil samples	1	2	3	4
pH, 1:1	7.85	8.30	6.66	8.27
OM, %	1.66	1.62	9.15	2.75
Soil texture	Sandy loam	Silt loam	Silt	Sandy loam
Sand, %	73.2	33.9	26.0	37.2
Silt, %	15.0	34.9	67.0	53.8
Clay, %	8.8	19.6	7.0	10.0

Residue and persistence of CCU in Chinese cabbage and soil in field

Plot *A*, *B*, *C*, *D*, *E*, *F* and *G* were designed to research the residue and persistence of CCU in vegetable and soil in field. Area of each plot was greater than $30m^2$. The persistence trials of CCU in Chinese cabbage and soil were carried out in plot *A* and *B* and residue trials in plots *C*, *D*, *E*, *F* and *G*. The rates and times of application in different plots are shown in Table 2. The CCU diluted 2500 fold with water were sprayed to the vegetable at regular intervals. The Chinese cabbage and soil were sampled at regular intervals to analyze the residue of CCU.

Extraction of sample

100 g of representative sample of Chinese cabbage were extracted with 250ml of dichloromethane in oscillator for 1 hour. The solid residues were extracted again with 150ml of dichloromethane. The combined extracts were dried by adding 20g of anhydrous Na_2SO_4 and then concentrated to dryness. The residue was dissolved in the solution with 1 ml methanol and CCU in methanol was analyzed by HPLC.

20g of representative sample of soil were extracted with 2×50ml of dichloromethane in oscillator for 1 hour. The combined extracts were dried by passing through an anhydrous Na_2SO_4 column and concentrated to dryness. The residues were dissolved in the solution with 1

ml methanol and CCU was analyzed by HPLC.

Analytical method

Shimadzu high performance liquid chromatograph (Japan) with stainless steel column 25cm × 4.6mm (i.d.) packed with ZORBAX-ODS was used to analyze the residue of CCU. The mobile phase consisting of a 80/20(v.v) mixture of methanol-water was pumped at 35°C with a flow rate of 1.0 ml/min.

The recoveries of CCU in Chinese cabbage were 88% for 0.1 – 1.0 ppm level and 86% in soil for 0.1 – 1.0 ppm level. The limit of detection of CCU in Chinese cabbage and soil by using the above procedure was equal to 0.01 ppm.

Table 2 Rates and times of CCU in different test plots

Year	Plot	Rate, g a.i./ha	Times	Date		
1988	*A*	300	1		10/12	
	B	300	1		10/17	
	C	150	3	9/1	9/19	10/12
	D	150	2	9/19	10/12	
	E	300	3	9/1	9/19	10/12
	F	300	2	9/19	10/12	
	G	0	0			
1989	*A*	300	1		9/10	
	B	300	1		9/1	
	C	150	3	9/10	9/22	10/7
	D	150	2	9/22	10/7	
	E	300	3	9/10	9/22	10/7
	F	300	2	9/22	10/7	
	G	0	0			

RESULTS AND DISCUSSIONS

Degradation of CCU in soil under laboratory condition

The experimental results of degradation of CCU in soils incubated at 28°C in the laboratory showed that the CCU is an unstable compound with half-life of 14.5, 7.2, 7.3 and 7.7 days in soil sample 1, 2, 3, and 4, respectively.

The persistence of CCU in Chinese cabbage and soils in field

The persistence experiments of CCU in Chinese cabbage and soils were carried out in plot *A* and *B*, respectively. The concentrations of CCU in Chinese cabbage and soil were monitored for a period of 28 or 56 days after the application of the pesticide. The results are shown in Table 3 and Table 4.

The disappearance of CCU in Chinese cabbage basically followed the first-order kinetics. The

concentration of CCU against time could be expressed by the equations:

$C=22.34e^{-0.0496t}$, $T_{1/2}=14.0$d, $r=-0.98$ (in the trial of 1988);

$C=8.00e^{-0.0524t}$, $T_{1/2}=13.2$d, $r=-0.99$ (in the trial of 1988).

where C is the concentration of CCU in Chinese cabbage (ppm); T is the time after application (days).

Table 3 Concentration of CCU in Chinese cabbage at different times after application (ppm)

Year	Sampling time, days							
	0	1	5	7	14	19	21	28
1988*	20.10			16.20	13.00	9.00		5.02
1989	7.83	7.74	6.00		4.0		1.40	1.81

* External leaves were studied

Table 4 Concentration of CCU in soils at different times after application (ppm)

Year	Sampling time, days												
	0	1	2	5	7	8	14	21	28	35	42	49	56
1988	0.36	0.34	0.33	0.31	0.27		0.24	0.21					
1989	3.70	3.07	3.05	2.99		1.40	0.74	0.50	0.36	0.28	0.26	0.32	0.28

In the same manner, the concentrations of CCU in soil at different times after application could be expressed also by following equations:

$C=0.35e^{-0.0256t}$, $T_{1/2}=27.0$d, $r=-0.98$ (in the trial of 1988);

$C=3.30e^{-0.0791t}$, $T_{1/2}=8.8$d, $r=-0.97$ (in the trial of 1989).

The results above indicated that CCU was not a long persistence pesticide. It disappeared fastly in Chinese cabbage and soil with half-life of 13.2–14.0 and 8.8–27.0 days, respectively. That was similar to the results of degradation of CCU in soil under laboratory conditions.

Some studies reported that the diflubenzuron, another important insect growth regulator was fairly stable in soil (Metcalf, 1975; Booth, 1977; Bull, 1978). Metcalf reported that diflubenzuron with Drummer soil-silty clay loam (17.4% moisture) was incubated in the laboratory. The recovery of parent diflubenzuron was greater than 99% after four weeks of incubation. Booth and Ferrell also reported that diflubenzuron was quite persistent under aerobic as well as anaerobic conditions. But Chapman (1985) reported that diflubenzuron was quite unstable in soil. Only 12% of parent diflubenzuron remained in soil after four weeks.

Residues of CCU in Chinese cabbage and soil in field after multiple application

In plot C, D, E, F and G residues of CCU in Chinese cabbage and soil were monitored after 2 or 3 applications of the pesticide at the rates of 150 or 300g a.i./ha as described in Table 1.

Samples of Chinese cabbage and soil were collected after the final application at intervals of 0,7,10,14,21, and 28 days. The content of CCU residues is listed in Table 5 and Table 6.

Table 5 The content of CCU residues in soil after multiple application* (ppm)

Year	Plot	Sampling time, days					
		0	7	10	14	21	28
1988	*C*	0.01	ND**		ND	ND	
	D	0.01	ND		ND	ND	
	E	0.02	ND		ND	ND	
	F	0.02	ND		ND	ND	
1989	*C*	0.10		0.07	0.04	0.01	ND
	D	0.03		0.02	0.01	ND	ND
	E	0.20		0.13	0.09	0.05	ND
	F	0.06		0.03	0.02	ND	ND

* Control samples contained less than 0.01 ppm of CCU

** ND≤0.01 ppm of CCU.

Tabe 6 Residue of CCU in Chinese cabbage after multiple application* (ppm)

Year	Plot	Sampling time, days					
		0	7	10	14	21	28
1988	*C*	1.49	1.32		0.93	0.65	
	D	1.26	0.98		0.66	0.45	
	E	1.75	1.31		1.07	0.73	
	F	1.41	0.93		0.88	0.68	
1989	*C*	2.41	0.83		0.43	0.17	0.04
	D	2.02	1.00		0.62	0.16	0.07
	E	7.93		2.64	0.78	0.36	0.07
	F	3.31		1.12	0.91	0.65	0.11

* Control samples contained less than 0.01 ppm of CCU

Table 5 shows that the residues of CCU in soil after multiple applications were considerable low. The maximum residue levels for CCU in soil were 0.09 ppm at 14th day, and 0.05ppm at 21th day and less than 0.01 ppm at 28th day after last application, respectively. The organic matter was 1.66% and pH 7.85 in the sample 1 tested in field. The experimental results showed that the CCU degradated fastly in basic soil.

Table 6 shows that multiple application of CCU did not result in high levels of CCU in Chinese cabbage. The maximum residue levels of CCU in Chinese cabbage were 1.32, 1.07 and

0.73 ppm in the trial of 1988, and 1.00, 0.91, 0.65, and 0.11 ppm in the trial of 1989 at 7, 14, 21 and 28th day after last application, respectively.

Based on the experimental results above and also toxicity test of CCU (Sun, 1982; Lu, 1983), 3 ppm as the maximum residue level for insecticide CCU in Chinese cabbage and 21 days as the preharvest interval would be recommended in the Northern China.

SUMMARY

The experimental results of degradation of CCU in soils under laboratory conditions and its residue and persistence in Chinese cabbage and soil in field in the Northern China showed that the insect growth regulator 1-(2-chlorobenzoyl)-3-(4-chlorophenyl) urea was not a long persistent pesticide. It degradated fastly in the vegetables and soils. The half-life of CCU were 13.2－14.0 days in Chinese cabbage and 8.8－27.0 days in soil, respectively. The maximum residue of CCU was 1.4 ppm in Chinese cabbage and 0.05 ppm in soil at 21th day after last spraying at the application rate of 150 or 300 g a.i./ha and with application frequencies of 2 or 3 times, 3 ppm as the maximum residue level for CCU in Chinese cabbage and 21 days as the preharvest interval for the vegetable treated with CCU were recommended.

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