

Effect of Processing on Reduction of λ -Cyhalothrin Residues in Tomato Fruits

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Received: 9 June 2011 / Accepted: 24 November 2011 / Published online: 15 December 2011
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Abstract Persistence behaviour of λ -cyhalothrin in tomato fruits was studied following application with recommended (15 g a.i. ha^{-1}) and double the recommended (30 g a.i. ha^{-1}) dosage of λ -cyhalothrin at fruiting stage under two different temperature conditions. Samples were collected on 0 (1 h after spray), 1, 3, 5, 7, 10 and 15 days after application. Effects of processing like washing, washing followed by boiling were studied to dislodge the residues of λ -cyhalothrin on tomato fruits. Residues were estimated by GC-ECD capillary system. Residues of λ -cyhalothrin persisted up to 7 days in tomato fruits. The half-life ($t_{1/2}$) periods were observed to be 2.07 and 1.88 days at room temperature and 2.35 and 2.02 days under refrigerated condition for single and double dose, respectively. The process of washing followed by boiling reduced the residues effectively (74–84%) whereas by washing only, residues could be reduced in the range of 37–40%. In samples under refrigerated condition, residues decreased slightly less than the samples stored under room temperature. Washing followed by boiling reduced the residues from 72 to 80% whereas only washing reduced the residues from 35 to 36%. In soil samples, residues of λ -cyhalothrin reached below detectable level of 0.005 mg kg^{-1} on 3rd and 7th day at single and double dose, respectively.

Keywords Tomato · λ -Cyhalothrin · Residues · Processing · Half-life period

Vegetables are an inseparable part of our daily diet and the vast reserves of growth promoting factors. Among vegetables, tomato occupies a key position with respect to their usage in human diet and cultivated area. Vegetables are prone to insect attack and thus get spoiled easily (Patel et al. 1994; Dikshit et al. 1980). To combat insect-pests of these crops and to achieve higher production, many pesticides are used. In this respect, λ -cyhalothrin (*S*)- α -cyano-3-phenoxy benzyl-(*Z*)-(1*R*, 3*R*)-3-(2-chloro-3,3,3-trifluoro prop-1-enyl)-2,2 dimethyl cyclopropane carboxylate, from an important group of synthetic pyrethroids, is widely used as domestic insecticide because of its relatively low toxicity and high knock down effect on insects. It has very high activity against a wide range of chewing and sucking insect pests, particularly lepidoptera, coleoptera and mites in fruits, vegetables, cereals, maize, cotton, wheat, pulses, oilseed and in public health as a vector control agent (Davey et al. 1992; Roberts et al. 1993; Dikshit et al. 2000; Mathirajan et al. 2000). The insecticide has been reported to be highly effective against termites and mites (Su et al. 1993; Davey et al. 1992). Now a days, the food safety issue induced by food contamination with reference to pesticide residue is becoming more and more important. The public attention is mainly focused on pesticide residues. Good knowledge of the pesticide fate in agriculture is necessary to properly assess human exposure and the environmental impact of contaminants, the concentrations of the pesticide residues in food commodities. Common household processing such as washing, peeling, cooking, blanching, concentrating can reduce residue levels in food and then reduce impact on human health (Abou-arab 1999; Byrne

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and Pinkerton 2004; Soliman 2001; Zohair 2001). Persistence behaviour and reduction of residues by processing is also affected by storage conditions (Gupta and Bhatnagar 1992; Malik et al. 1998; Gill et al. 2001). Keeping these facts in view, present study was carried out to know the persistence and effect of processing on reduction of λ -cyhalothrin residues in tomato fruits at two different temperature i.e. at room temperature and under refrigerated (4°C) conditions.

Materials and Methods

The field trials for the determination of λ -cyhalothrin residues in tomato (*Lycopersicon esculentum* L.) (variety HS-102) was conducted in the research field of Entomology, CCS Haryana Agricultural University, Hisar during April–May 2010 in separate plots measuring 3 m × 3 m each in three replicates with randomized block design (RBD) and two treatments along with control. The supervised field plots, received all the routine agronomic practices and the crop was sprayed with commercial λ -cyhalothrin (Karate 2.5 EC) at recommended (15 g a.i. ha^{-1}) (T_1) and double the recommended (30 g a.i. ha^{-1}) (T_2) dose.

For residue analysis, the treated samples of tomato fruits were drawn at the periodic intervals of 0 (1 h), 1, 3, 5, 7, 10, 15 days after treatment and processed on the same day. These samples were used to study the persistence and effect of processing, viz. washing (under tap water for 30 s) with gentle rotation by hand as described by Walter et al. (2000) and washing followed by boiling at two different temperature, i.e. room temperature and under refrigerated (4°C) condition.

Soil samples of 0–15 cm soil depth from each treatment were collected with same schedule as of tomato fruits i.e. on 0 (1 h), 1, 3, 5, 7, 10 and 15 days after spray, shade dried, crushed in pestle and mortar and sieved through 2 mm sieve.

The residues were extracted from all the samples of tomato using the method of Jayakrishnan et al. (2005). Representative 25 g chopped sample was extracted with 100 mL acetone on the mechanical shaker for 1 h. The extract was filtered, transferred directly to separatory funnel, diluted with 10% aqueous solution of NaCl and partitioned thrice (70, 50, 30 mL) with dichloromethane (DCM) each time with vigorous shaking. The DCM phases were combined, passed through anhydrous sodium sulphate and concentrated to 5 mL using rotary vacuum evaporator.

The tomato fruit extract was cleaned by column chromatography. Glass column (60 cm × 2.2 mm i.d.) was packed with 5 g Florisil in between two layers of anhydrous sodium sulphate. The column was eluted with

125 mL hexane:acetone (9:1 v/v). The extract was concentrated to near dryness on rotary vacuum evaporator and made the final volume to 2 mL in *n*-hexane.

Soil samples were processed as per method of Kumari et al. (2008). Ground, sieved and dry representative (15 g) of soil sample was mixed thoroughly with 0.3 g activated charcoal, 0.3 g Florisil and 10 g of anhydrous sodium sulphate. The mixture was packed compactly in a glass column (60 cm × 22 mm) in between two layers of anhydrous sodium sulphate. Pesticide residues were eluted with 125 mL of hexane: acetone (9:1 v/v) mixture at flow rate of 2–3 mL/min. Concentrated the eluate on flash evaporator and made the final volume to 2 mL for GC analysis.

The λ -cyhalothrin residues were determined by gas liquid chromatography (GC) model Shimadzu Model 2010 equipped with ECD (Ni⁶³), split injection system and fused capillary column (SPB-5) 30 m × 0.32 mm I.D., 0.25 μ m film thickness of polysiloxane (5% diphenyl/95% dimethyl). GC operating conditions were: Carrier gas flow, 60 mL min^{-1} , injector temperature 280°C, oven temperature programme was 150°C (5 min) increasing @ 8°C min^{-1} up to 190°C (2 min), further increasing @ 15°C min^{-1} up to 280°C (10 min). The recovery experiments were carried out at 0.25 mg kg^{-1} and 0.50 μ g g^{-1} fortification levels of λ -cyhalothrin. In tomato, percent recoveries ranged between 87 and 92% and in soil from 97 to 101%. The limit of quantification (LOQ) was 0.010 and 0.005 mg kg^{-1} in tomato and soil, respectively.

Results and Discussion

The residue data of persistence behaviour of λ -cyhalothrin for two doses, i.e. 15 g a.i. ha^{-1} (T_1) and 30 g a.i. ha^{-1} (T_2) under room temperature and under refrigerator condition have been shown in Tables 1 and 2. The initial deposits of λ -cyhalothrin at single and double dosages in tomato crop, on 0 day (1 h after application) were 0.144 and 0.354 mg kg^{-1} and it reached to 0.086 and 0.206 mg kg^{-1} on 3rd day by showing 40.27% and 41.80% dissipation, respectively under room temperature. The data show that the residues were dissipated with time and reached below detectable level of 0.010 mg kg^{-1} with in 7 days in both the doses. Under refrigerated conditions, dissipation was slightly low as compared to room temperature. Under this condition, initial deposit of 0.144 and 0.354 mg kg^{-1} reached to the levels of 0.090 and 0.210 mg kg^{-1} after 3 days showing dissipation of 37.50 and 40.67 per cent in single and double dose, respectively. After 10 days, no residues were detected in the marketable fruits in any dose and both the conditions. The residues dissipated with half-life period of 2.07 and 1.88 days at

Table 1 Persistence of λ -cyhalothrin residues in tomato under room temperature

Days after treatment	Residue (mg kg ⁻¹) ^a			
	T_1 (15 g a.i. ha ⁻¹)		T_2 (30 g a.i. ha ⁻¹)	
	Average \pm SD	% Dissipation	Average \pm SD	% Dissipation
0	0.144 \pm 0.057	–	0.354 \pm 0.069	–
1	0.114 \pm 0.005	20.83	0.276 \pm 0.045	22.00
3	0.086 \pm 0.051	40.27	0.206 \pm 0.104	41.80
5	0.052 \pm 0.050	63.88	0.124 \pm 0.095	64.97
7	0.011 \pm 0.051	92.56	0.020 \pm 0.010	94.35
10	BDL	–	BDL	–
15	BDL	–	BDL	–

Correlation coefficient $r = -0.9356$
 Regression equation = $1.2470 + 0.1455x$
 $t_{1/2} = 2.07$ days

Correlation coefficient $r = -0.9242$
 Regression equation = $1.6545 + 0.1609x$
 $t_{1/2} = 1.88$ days

^a Average of three replicates

BDL below detectable level

Table 2 Persistence of λ -cyhalothrin residues in tomato under refrigerated conditions

Days after treatment	Residue (mg kg ⁻¹) ^a			
	T_1 (15 g a.i. ha ⁻¹)		T_2 (30 g a.i. ha ⁻¹)	
	Average \pm SD	% Dissipation	Average \pm SD	% Dissipation
0	0.144 \pm 0.057	–	0.354 \pm 0.069	–
1	0.119 \pm 0.005	17.36	0.282 \pm 0.066	20.33
3	0.090 \pm 0.063	37.50	0.210 \pm 0.100	40.67
5	0.059 \pm 0.066	59.02	0.130 \pm 0.062	63.27
7	0.015 \pm 0.045	89.58	0.025 \pm 0.006	92.93
10	BDL	–	BDL	–
15	BDL	–	BDL	–

Correlation coefficient $r = -0.9370$
 Regression equation = $1.2374 + 0.1282x$
 $t_{1/2} = 2.35$ days

Correlation coefficient $r = -0.9299$
 Regression equation = $1.6441 + 0.1492x$
 $t_{1/2} = 2.02$ days

^a Average of three replicates

BDL below detectable level

room temperature and 2.35 and 2.02 days under refrigerated conditions at single and double dose, respectively, following biphasic first order kinetics (Figs. 1, 2). Under refrigerated conditions, dissipation was relatively less as compared to room temperature. Similar observations have been reported by Mourkidou et al. (1994); Kawara et al. (1973) and Malik et al. (1998).

Tomato fruits were subjected to processing like washing and washing followed by boiling in order to investigate the reduction of residues. The data on effect of processing on reduction of λ -cyhalothrin residues under room temperature and refrigerated conditions are shown in Tables 3 and 4. It has been found that washing followed by boiling was found to be more effective than washing only in reducing

the residues under both conditions of temperature. The percent reduction in residues due to washing followed by boiling ranged from 57.63 to 74.41% at single dose and from 59.88 to 83.87% at double dose at room temperature whereas respective reduction under refrigerated conditions were 53.47–72.22% at single dose and from 56.21 to 80.00% at double dose. Reduction of residues by washing ranged from 37.50 to 40.11% under room temperature and from 34.77 to 36.44% under refrigerated conditions in both the doses. It is clear from the data that reduction of residues by processing was higher at room temperature than under refrigerated conditions. Present results are in agreement with earlier reports (Jayakrishnan et al. 2005) in which 30.0–39.0% reduction of λ -cyhalothrin residues on tomato

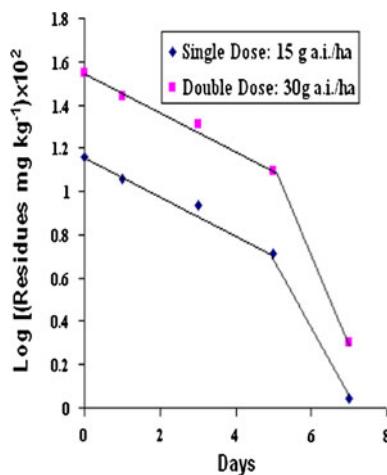


Fig. 1 Linear plot for first order kinetics of λ -Cyhalothrin at room temperature

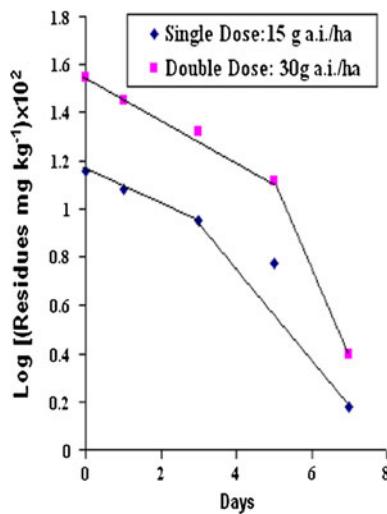


Fig. 2 Linear plot for first order kinetics of λ -Cyhalothrin at refrigerated condition

was observed while in washing plus cooking reduction was from 67.54 to 76.69%. (Sunderajan 1983) reported that 90% permethrin dissipated from tomato fruits within 1 week under field conditions. Under refrigerated conditions the initial deposits of 0.144 and 0.354 $\mu\text{g g}^{-1}$ at single and double dosages respectively, dissipated by 17.36% after 1 day, 37.50% in 3 day and 89.58% in 7th day at single dose while at double dose the percent dissipation was 20.33, 40.67, 92.93 on 1, 3 and 7th day showing less reduction under cold storage conditions. Similar observations have been made by Gupta and Bhatnagar (1992) also. During washing of tomato sample stored under cold condition reduced the residues by about 34.77% in 0 day and 26.89% and 22.22% on 1st and 3rd day followed by washing plus cooking under cold storage conditions the residues were reduced up to 80% on 5th day. Slow

Table 3 Effect of processing on λ -cyhalothrin residues in tomato under room temperature

Days	Residues (mg kg ⁻¹) ^a			T ₁ (15 g a.i. ha ⁻¹)			T ₂ (30 g a.i. ha ⁻¹)			Washing + boiling		
	Initial residues \pm SD			Washing			Washing + boiling			Washing		
	Residue \pm SD	% Reduction	Residue \pm SD	% Reduction	Residue \pm SD	% Reduction	Residue \pm SD	% Reduction	Residue \pm SD	% Reduction	Residue \pm SD	% Reduction
0	0.144 \pm 0.057	0.090 \pm 0.037	37.50	0.061 \pm 0.033	57.63	0.354 \pm 0.069	0.212 \pm 0.017	40.11	0.142 \pm 0.046	59.88	0.142 \pm 0.046	59.88
1	0.114 \pm 0.005	0.080 \pm 0.012	29.82	0.037 \pm 0.020	67.54	0.276 \pm 0.045	0.188 \pm 0.042	31.88	0.092 \pm 0.051	66.66	0.092 \pm 0.051	66.66
3	0.086 \pm 0.050	0.066 \pm 0.055	23.25	0.022 \pm 0.001	74.41	0.206 \pm 0.104	0.155 \pm 0.017	24.75	0.048 \pm 0.044	76.69	0.048 \pm 0.044	76.69
5	0.052 \pm 0.050	BDL	—	BDL	—	0.124 \pm 0.095	0.101 \pm 0.031	18.54	0.020 \pm 0.027	83.87	0.020 \pm 0.027	83.87
7	0.041 \pm 0.051	BDL	—	BDL	—	0.020 \pm 0.010	BDL	—	BDL	—	BDL	—
10	BDL	BDL	—	BDL	—	BDL	BDL	—	BDL	—	BDL	—
15	BDL	BDL	—	BDL	—	BDL	BDL	—	BDL	—	BDL	—

^a Average of three replicates

Table 4 Effect of processing on λ -cyhalothrin residues in tomato under refrigerator condition

Days	Residues (mg kg ⁻¹) ^a						T_2 (30 g a.i. ha ⁻¹)	
	T_1 (15 g a.i. ha ⁻¹)			Washing + boiling				
	Initial Residues \pm SD	Washing	Residue \pm SD	% Reduction	Initial Residues \pm SD	Washing		
0	0.144 \pm 0.057	0.094 \pm 0.045	34.77	0.067 \pm 0.044	53.47	0.354 \pm 0.019	0.225 \pm 0.033	
1	0.119 \pm 0.006	0.087 \pm 0.058	26.89	0.043 \pm 0.027	63.86	0.282 \pm 0.037	0.199 \pm 0.027	
3	0.090 \pm 0.060	0.070 \pm 0.047	22.22	0.025 \pm 0.019	72.22	0.210 \pm 0.010	0.162 \pm 0.017	
5	0.059 \pm 0.037	BDL	—	BDL	—	0.130 \pm 0.006	0.110 \pm 0.011	
7	0.015 \pm 0.031	BDL	—	BDL	—	0.025 \pm 0.006	BDL	
10	BDL	BDL	—	BDL	—	BDL	—	
15	BDL	BDL	—	BDL	—	BDL	—	

^a Average of three replicates**Table 5** Residues (mg kg⁻¹) of λ -cyhalothrin in soil

Days	Single dose (15 g a.i. ha)		Double dose (30 g a.i. ha)	
	Average ^a residues \pm SD	% Reduction	Average ^a residues \pm SD	% Reduction
0(1 h)	0.011 \pm 0.011	—	0.021 \pm 0.005	—
1	0.007 \pm 0.019	36.36	0.014 \pm 0.012	33.33
3	0.005 (BDL) \pm 0.015	54.54	0.011 \pm 0.023	47.61
5	BDL	100	0.006 \pm 0.003	71.42

^a Average of three replicates

reduction of alphamethrin residues under refrigerated conditions in cauliflower, brinjal and tomato have been reported by Malik et al. (1998); Gill et al. (2001).

In soil, the initial deposits of 0.011 and 0.021 mg kg⁻¹ (Table 5) dissipated by 36.36 and 33.33% on 1 day at single and double dosages, respectively and become below detectable level (BDL) of 0.005 mg kg⁻¹ after 3rd day in single dose and after 5th day in double dose. Low residues in soil may be attributed to bushy crop of tomato resulting very less fall of sprayed insecticide on soil.

Acknowledgments The authors wish to express their gratitude to the Head, Department of Entomology for providing research facilities.

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