

Dissipation and Decontamination of Bifenthrin Residues in Tomato (*Lycopersicon esculentum* Mill)

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Abstract A field experiment was conducted at the Research Farm of CCS HAU, Hisar to study the dissipation and decontamination behavior of bifenthrin on tomato crop following the application of 25 g a.i ha⁻¹ (T₁) and 50 g a.i ha⁻¹ (T₂). Samples were collected periodically on the sampling days after applications. Residues were reached below detectable level of 0.005 mg kg⁻¹ on 10th day after application showing half-life period of 1.83 and 2.05 days at room temperature and 2.02 and 2.32 days under refrigerated condition for single and double dose, respectively. Processing was found effective in reducing the residues of bifenthrin in tomato fruits. Maximum reduction (42.10–45.23 %) was observed by washing + boiling followed by washing (16.66–19.04 %). Reduction was slightly less when samples were stored under refrigerated conditions as compared to room temperature conditions.

Keywords Tomato · Bifenthrin · Residues · Processing · Half-Life Period · Soil

Pesticides are used globally for the protection of food, fiber, human health and comfort. Food is the basic necessity of life and food contaminated with toxic pesticides is associated with severe effects on the human health. Hence it is pertinent to explore strategies that address the situation of food safety especially for the developing countries

where pesticidal contamination is widespread due to indiscriminate usage. Scientists and food processors have long been interested in the effect of commercial processing on reduction of pesticide residues in food and it was observed that pesticide residues in plant produce are reduced by processing or some household preparations like washing, peeling, cooking etc. (Dikshit et al. 2003).

Tomato (*Lycopersicon esculentum* Mill.) is most widely grown vegetable in the world for several hundred years and known as apple of poor men. The crop is susceptible to attack by a number of insect pests from the time plant first emerges in the seed bed until harvest. In the field, it is mainly attacked by jassids, aphids, tobacco caterpillar, flea beetles, leaf miners, and fruit borers. Bifenthrin, ((2-methyl-1, 1-biphenyl-3-yl)-methyl-3-(2-chloro-3, 3, 3-trifluoro-1-propenyl)-2, 2-dimethyl cyclopropanecarboxylate, is an insecticide with contact and stomach poison. It has shown good bioefficacy against insect pests of brinjal (Sudhakar et al. 1998) and tomato (Rushtapakornchai and Petchwichit 1996).

The present study was designed to investigate bifenthrin residues and effect of processing on reduction of its residues in tomato fruits at two different temperatures i.e. at room temperature and under refrigerated (4°C) conditions.

Materials and Methods

In a field experiment, bifenthrin (Brigade 2.5EC) @ single (25 g a.i. ha⁻¹) and double dose (50 g a.i. ha⁻¹) was applied on the tomato crop (variety HS 102) with Knap Sap sprayer in plots of 3 × 3 m size along with a control plot.

Tomato fruits and soil samples in triplicate were collected randomly from each plot at 0 (1 h), 1, 3, 5, 7, 10 and

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15 days after applications for dissipation study. Samples were collected randomly and periodically from each plot in triplicate along with control. Soil samples were collected from top 15 cm of soil profile with the help of soil auger, shade dried, crushed in pestle and mortar and sieved through 2 mm sieve.

Tomato samples collected from field were analyzed at three stages i.e. raw, after washing and washing followed by boiling under two conditions i.e. room temperature and refrigerated conditions (4°C) to determine the residues of bifenthrin. The tomato samples were divided into two lots one for room temperature conditions and the second one kept in refrigerator. Further, the samples of both sets of conditions were again divided into three portions, one portion was processed as such second after washing and third one after washing followed by boiling/cooking.

Washing was performed with gentle rotation by hand under normal tap water for 30 s, following the method of Walter et al. (2000) and blotted dry with a paper towel and divided into two parts. For boiling, 25 g representative samples of tomato, 10 mL water was added and boiled till softness of tomato pieces for both sets of conditions.

Extraction and clean-up was performed as per method of Jayakrishnan et al. (2005). Representative 25 g finely chopped sample was extracted with 100 mL acetone by shaking it on mechanical shaker for 1 h. The extract was filtered and partitioned thrice with dichloromethane. The organic layer was collected and concentrated on rotary vacuum evaporator up to 10 mL. For clean-up, glass column (60 cm × 22 mm i.d.) was packed compactly with activated Florisil and activated charcoal (5:1 w/w) in

between two layers of anhydrous sodium sulphate. Pre wetting was done with n-hexane, loaded the concentrated extract in the column and eluted with 125 mL hexane: acetone (9:1 v/v). Concentrated the eluate on vacuum evaporator followed by gas manifold evaporator. Final volume was made to 2 mL in n-hexane for GC analysis.

Soil samples were processed as per method of Kumari et al. (2008). Ground, sieved and dry representative (15 g) of soil mixed with activated charcoal and Florisil (0.3 g each) 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. Residues were eluted with hexane: acetone (9:1 v/v) mixture. The eluate was concentrated on flash evaporator and made the final volume to 2 mL for GC analysis.

Bifenthrin residues were quantified on Shimadzu 2010, a gas chromatograph (GC) equipped with fused capillary column, HP-1 of 30 m × 0.32 mm i.d, 0.25 µm film thickness of polysiloxane (5 % diphenyl/95 % dimethyl) and electron capture detector (ECD). The operating parameters of GC were: carrier gas flow, 60 mL min⁻¹, injector temperature 280°C, oven temperature programme was 150°C (5 min) increasing @ 8°C min up to 190°C (2 min.), further increased @ 15°C min⁻¹ up to 280°C (10 min) with split ratio 1: 10. The retention times observed for bifenthrin was 18.480 min. The percent recoveries of bifenthrin in tomato were 90.00 and 92.23 and in soil were 94.13 and 96.14 at 0.10 and 0.25 mg kg⁻¹ levels, respectively. Limit of detection was 0.002 and limit of determination/quantitation was 0.005 mg kg⁻¹.

Table 1 Residues (mg kg⁻¹)^a of bifenthrin in tomato fruits under room temperature

Days after treatment	Residue (mg kg ⁻¹)				Mean
	T ₁ (25 g a.i.ha ⁻¹)		T ₂ (50 g a.i.ha ⁻¹)		
	Average ± SD	% Dissipation	Average ± SD	% Dissipation	
0	0.107 ± 0.012	–	0.234 ± 0.032	–	0.171
1	0.086 ± 0.003	19.62	0.193 ± 0.020	17.52	0.140
3	0.038 ± 0.002	64.48	0.087 ± 0.012	62.82	0.063
5	0.018 ± 0.022	83.15	0.042 ± 0.002	82.05	0.030
7	0.008 ± 0.001	92.52	0.021 ± 0.012	91.88	0.015
10	BDL	–	0.009 ± 0.022	97.86	0.005
15	–	–	BDL	–	0.000
Mean	0.043		0.098		
Correlation coefficient $r = -0.9984$			Correlation coefficient $r = -0.9980$		
Regression equation $y = 2.0646 - 0.1638x$			Regression equation $y = 2.3841 - 0.1465x$		
$t_{1/2} = 1.83$ day			$t_{1/2} = 2.05$ day		

CD ($p = 0.05$) for days = 0.001 for dose = 0.001 for days × dose = 0.002

For regression equation, [Residues (mg kg⁻¹) × 10³] is taken

^a Average of three replicates; BDL below detectable level (0.005 mg kg⁻¹)

Table 2 Residues (mg kg^{-1})^a of bifenthrin in tomato fruits under refrigerated conditions

Days after treatment	Residue (mg kg ⁻¹) ^a				Mean
	T ₁ (25 g a.i.ha ⁻¹)		T ₂ (50 g a.i.ha ⁻¹)		
	Average ± SD	% Dissipation	Average ± SD	%Dissipation	
1	0.090 ± 0.011	15.88	0.201 ± 0.007	14.10	0.146
3	0.041 ± 0.005	61.68	0.092 ± 0.019	60.68	0.067
5	0.022 ± 0.023	79.43	0.050 ± 0.033	78.63	0.036
7	0.010 ± 0.004	90.65	0.029 ± 0.014	87.60	0.020
10	0.007 ± 0.002	93.45	0.013 ± 0.010	94.44	0.010
15	BDL	–	BDL	–	0.000
Mean	0.034		0.077		
Correlation coefficient $r = -0.9974$			Correlation coefficient $r = -0.9975$		
Regression equation = $2.0656 - 0.1493x$			Regression equation = $2.3784 - 0.1292x$		
$t_{1/2} = 2.02$ day			$t_{1/2} = 2.32$ day		

CD ($p = 0.05$) for days = 0.001 for dose = 0.001 for days \times dose 0.002

For regression equation, [Residues (mg kg^{-1}) $\times 10^3$] is taken

^a Average \pm SD of three replicates

Result and Discussion

Residue data and percent dissipation of bifenthrin at single and double dose in tomato under two sets of conditions are given in Tables 1 and 2 (Figs. 1 and 2). The average initial deposits estimated in tomato were found to be 0.107 and 0.234 mg kg^{-1} on 0 day after application, respectively. These residues level dissipated to an extent of 0.086 and 0.193 mg kg^{-1} on 1 day after application at single and double dose, respectively. The per cent dissipation observed on 7th day was 92.52 and 91.88 and thereafter reached below detectable level of 0.005 mg kg^{-1} on 10 and 15 days after application in single and double dose, respectively under room conditions. Under refrigerated conditions, dissipation was slightly low as compared to room temperature. Under this condition, average initial deposits in single and double dose were 0.090 and

0.201 mg kg^{-1} one day after application showed per cent dissipation of 15.88 and 14.10 at the respective doses. After 15 days, no residues were detected in the marketable fruits in any dose and in both the conditions. Residue data were subjected to statistical analysis for computation of regression equations, half-life ($t_{1/2}$) values and per cent degradation. The residues dissipated with half-life period of 1.83 and 2.05 days at room temperature and 2.02 and 2.32 days under refrigerated conditions at lower and higher dose following first order kinetics. Under refrigerated conditions, dissipation was relatively less as compared to room temperature. Similar type of observation have been reported by Chauhan et al. (2012), Gill et al. (2001), Malik et al. (1998), Mourkidou et al. (1994).

Tomato fruits were subjected to processing i.e. washing and washing followed by boiling in order to investigate the reduction of residues as shown in Tables 3 and 4. The percent reduction due to washing followed by boiling ranged from 36.44 to 42.10 and 38.04 to 45.23 at lower and higher dose under room conditions whereas 37.77–40.90 %

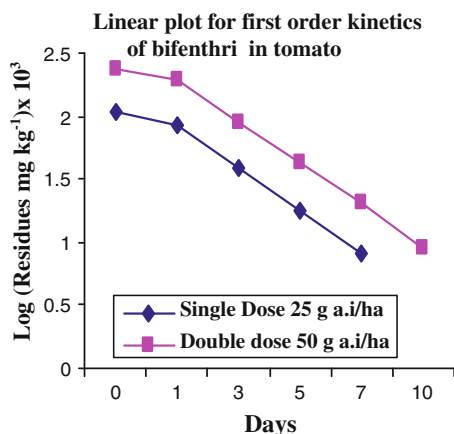
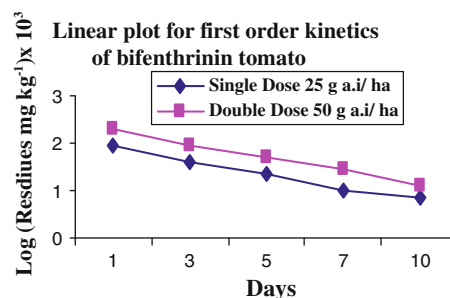
**Fig. 1** Under room temperature**Fig. 2** Refrigerated conditions (RC)

Table 3 Effect of processing on bifenthrin residues in tomato under room temperature

Residues (mg kg ⁻¹) ^a		T ₂ (50 g a.i.ha ⁻¹)					
Days	T ₁ (25 g a.i.ha ⁻¹)	Initial residues ± SD		Washing + boiling		Washing	
		Residue ± SD	% Reduction	Residue ± SD	% Reduction	Residue ± SD	% Reduction
0	0.107 ± 0.011	0.073 ± 0.014	31.77	0.068 ± 0.021	36.44	0.152 ± 0.004	35.04
1	0.086 ± 0.023	0.064 ± 0.036	25.58	0.052 ± 0.017	39.53	0.140 ± 0.002	27.46
3	0.038 ± 0.034	0.030 ± 0.011	21.05	0.022 ± 0.006	42.10	0.067 ± 0.013	22.98
5	0.018 ± 0.002	0.015 ± 0.005	16.66	BDL	–	0.034 ± 0.009	19.04
7	0.008 ± 0.017	BDL	–	–	–	BDL	–
10	BDL	–	–	–	–	–	–
15	–	–	–	–	–	–	–

^a Average ± SD of three replicates; BDL: 0.005 mg kg⁻¹**Table 4** Effect of processing on bifenthrin residues in tomato under refrigerated condition

Residues (mg kg ⁻¹) ^a		T ₂ (50 g a.i.ha ⁻¹)					
Days	T ₁ (25 g a.i.ha ⁻¹)	Initial residues ± SD		Washing + Boiling		Washing	
		Residue ± SD	% Reduction	Residue ± SD	% Reduction	Residue ± SD	% Reduction
1	0.090 ± 0.033	0.069 ± 0.003	23.33	0.056 ± 0.017	36.44	0.150 ± 0.110	25.37
3	0.041 ± 0.020	0.033 ± 0.011	19.51	0.025 ± 0.005	39.53	0.073 ± 0.035	20.65
5	0.022 ± 0.017	0.019 ± 0.004	13.63	0.013 ± 0.002	42.10	0.041 ± 0.021	18.00
7	0.010 ± 0.004	BDL	–	BDL	–	0.025 ± 0.007	17.24
10	0.007 ± 0.002	–	–	–	–	BDL	–
15	BDL	–	–	–	–	BDL	–

^a Average ± SD of three replicates; BDL: 0.005 mg kg⁻¹

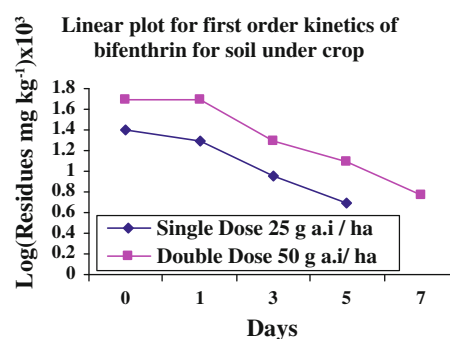
Table 5 Persistence of bifenthrin in soil under tomato crop

Days	Single dose (25 g a.i./ha)		Double dose (50 g a.i./ha)	
	Average ^a Residues (mg kg ⁻¹) ± SD	% Dissipation	Average ^a Residues (mg kg ⁻¹) ± SD	% Dissipation
0 (1 h)	0.026 ± 0.012	–	0.058 ± 0.011	–
1	0.021 ± 0.001	19.20	0.050 ± 0.013	13.79
3	0.009 ± 0.015	65.38	0.021 ± 0.001	63.79
5	0.005 ± 0.011	80.76	0.013 ± 0.012	77.58
7	BDL		0.006 ± 0.001	89.65
Correlation coefficient $r = -0.9955$			Correlation coefficient $r = -0.9951$	
Regression equation $y = 1.4329 - 0.1490x$			Regression equation $y = 0.7921 - 0.1427x$	
$t_{1/2} = 2.02$ day			$t_{1/2} = 2.10$ day	

^a Average ± SD of three replicates; BDL 0.005 mg kg⁻¹

and 38.80–42.00 % reduction was observed under refrigerated conditions for both the treatments. It has been found that washing followed by boiling was found to be more effective than washing in reducing the residues under two sets of conditions. However, by washing, residues were reduced in the range of 31.77–16.66 % at lower and 35.04–19.04 % at higher dose under room conditions. The percent reduction was 23.33–13.63 and 25.37–17.24 under refrigerated conditions for both the doses. The most interesting conclusions of these studies are that the rinsability of a pesticide is not always correlated with its water solubility (Cengiz et al. 2007; Boulaid et al. 2005; Angioni et al. 2004; Krol et al. 2000). The reduction of residues by processing was higher at room temperature than under refrigerated conditions as it is clear from the data. The results are in line with some other recent findings of Jayakrishnan et al. (2005) observed 30.0–39.0 % reduction of λ -cyhalothrin residues on tomato while in washing plus cooking, reduction was from 67.54–76.69 %. Kwon et al. (2009) did not observed significant change in residues of bifenthrin in spinach, chard and mellow by boiling whereas 58.0–64.0 % reduction was observed by washing. Zhang et al. (2007) reported that stir frying at 100°C for 5 min reduced the residues of cypermethrin by 84.7 percent in cabbage. Chauhan et al. (2012) reported that reduction of λ -cyhalothrin residues in tomato fruits was comparatively less under refrigerated condition than room temperature conditions. Slow reduction of alphanethrin residues under refrigerated conditions in cauliflower, brinjal and tomato have been reported by Malik et al. (1998) and Gill et al. (2001).

Residue data and percent dissipation of bifenthrin in soil under field conditions at two treatments are given in Table 5 (Fig. 3). The average initial deposits estimated in soil were found to be 0.026 and 0.058 mg kg⁻¹, respectively, following application of bifenthrin @ 25 and 50 g a.i./ha. The per cent reduction observed was 19.20, 65.38

**Fig. 3** Dissipation kinetics of bifenthrin for soil

and 80.76 at lower dose and 13.79, 63.79, 77.58 and 89.65 at higher dose on 1,3,5 and 7 days after application. The half-life periods were 2.02 and 2.10 days at single and double dose, respectively, following the first order kinetics.

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