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Residue Behavior of Fluvalinate in Chili (*Capsicum annuum* L.) under Indian Climatic Condition

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Chili (Capsicum annuum L.) is widely used in India as a vegetable crop and suffers heavily from the ravages of pests like borer,hoppers, polyphagous mites, aphids, jassids, thrips etc. throughout its growth stage. For controlling these pests, fluvalinate *[*(R,S) $-\alpha-$ cyano - 3 - phenoxybenzyl (R) 2 - (2 - chloro - (trifluromethyl) anilino - 3 methyl butanoate*]* , a synthetic pyrethroid is used extensively now-a-days during the cultivation of this crop. Studies on the persistence and degradation of this insecticide in different plants in different climatic conditions have been reported (Agnihotri et. al. 1986; Gopal et. al. 1987; Srivastava et. al. 1987; Fitch et. al. 1988; Mukherjee and Gopal 1990; Verma and Dixit 1990; Gupta et. al. 1990; Agnihotri et. al. 1992). Since this insecticide is toxic and used at 50% fruit developing stage, toxic residues may remain in the crop and pose a serious health hazard to the consumers. Therefore, this experiment was conducted to evaluate the persistence of fluvalinate on chili foliage, fruit and cropped-soil which may be the ultimate sink of pesticides.

MATERIALS AND METHODS

Chili (Pusa Jawala) was grown at University Research Farm of B.C.K.V. Kalyani (Sand 51%, Silt 31%, Clay 18% ; pH 6.5; Organic Carbon 0.5%) during the rainy season in randomised block design with 5 replications for each treatment. Fluvalinate 20 AF was applied at 50% fruit formation stage at a dose of 20 g a.i./ha with water @ 500 L/ha. For the control, water was sprayed @ 500 L/ha. The detail of the management practices are given below :

Net plot size	:	15 sq m
Spacing	:	60 x 45 sq cm
Fertilizer applied	:	N (110 kg/ha), P (70 kg/ha) and K (75 kg/ha)
Maximum temperature	:	34.3 °C
Minimum temperature	:	23.5 °C
Relative humidity	:	63 – 98%
Rainfall	:	0 – 23.4 mm

Commercial formulation of fluvalinate (20 AF) was analysed by GLC and found to contain 19.8%. Thus for field application, the dilution of fluvalinate was made accordingly.

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Chili foliage, fruit and cropped-soil samples from each replication were drawn separately at 0 (2 hr after spray), 1, 3, 5, 7 and 10 d after fluvalinate application. Soil samples were collected with the help of a soil auger from a depth of 0-12 cm at different spots in each plot and representative samples of 50 g were taken by quatering.

50 g foliage and fruit samples were chopped into small pieces and tumbled overnight in a glass jar with 100 mL n-hexane - acetone. The material was then filtered by light suction on a Büchner funnel. The remnants were then transferred back to the blender, re-extracted with 2 x 50 mL of the same solvent and the contents were filtered again under suction. The filtrates were combined. 50 g soil was taken into a 250 mL conical flask with 100 mL n-hexane - acetone (1:1) followed by to-and-fro shaking (6 hr) by a mechanical shaker. It was then filtered on a Büchner funnel and washed thoroughly with 2 x 50 mL of the same solvent mixture.

The combined filtrate obtained from foliage, fruit and soil samples was quantitatively transferred to a separatory funnel with acetone rinse, diluted with 250 mL saturated aqueous solution of NaCl. The upper n-hexane fraction was separated after vigorous shaking and it was collected after passing through anhydrous sodium sulphate. The aqueous layer was re-extracted with n-hexane (2x50 mL). The n-hexane fraction was concentrated to 20 mL by Kuderna Danish evaporator and dried over anhydrous sodium sulphate. The concentrated extracts of the samples were cleaned up by adding 1 g activated charcoal and 2 mL acetone. Mixture was allowed to stand for 2 min with occasional stirring. It was then filtered through Whatman No. 41 filter paper and washed with 3x15 mL n-hexane - acetone (9:l). Filtrate was concentrated and volume was made up to 5 mL with n-hexane.

Fruits with 0, 1 and 3 d residues were washed and boiled separately to see the possible mitigation of residues.

Fluvalinate residues were measured by gas-liquid chromatograph (model 5890A, Hewlett Packard, U.S.A.) equipped with ECD and coupled with 3392A integrator. Aliquots (l-10 µL) of residue extracts were injected with borosilicate glass column (6'x1/4" i.d.) packed with 3% DC 200 on 80-100 mesh chromosorb W.A.W. at a column temperature of 200 °C. The injection and detector temperature were 275 °C each. The flow of carrier-gas N_2 was 65 mL/min. The retention time of fluvalinate was 5.67 min. The response was linear in the range between 0.2 - 1.0 ng.

The efficiency of extraction, clean up and estimation procedures were checked from recovery experiments for fluvalinate by fortifying the respective samples. The recovery experiments for fluvalinate treated foliage, fruit and soil ranged from 81 - 92%. The rate of dissipation was worked out by determining the $T_{1,2}$ (Hoskins 1961).

RESULTS AND DISCUSSION

Fluvalinate residues on chili foliage, fruit and cropped-soil were 1.11, 0.35 and 1.06 ppm that dissipated to a non-detectable level

after 10 d of application (Table 1). The progressive loss of fluvalinate residues was 94-99% after 7 d of application. The half- life values in foliage, fruit and soil were in the range of l-l.7 d.

Table 1. Fluvalinate residues in/on chili foliage, fruit and soil

Days after treatment	Fluvalinate residues(ppm)*	Regression equation	$T_{1/2}$ (d)
	FOLIAGE		
0	1.11 ± 0.0	Y = 3.16 - 0.17X	1.73
1	1.0 ± 0.01 (9.91)		
3	0.60 ± 0.03 (45.94)		
5	0.23 ± 0.03 (79.28)		
7	0.06 ± 0.01 (94.60)		
10	N.D.		
	FRUIT		
0	0.35 ± 0.11	Y = 2.59 - 0.17X	1.70
1	0.28 ± 0.11 (20.0)		
3	0.13 ± 0.04 (62.80)		
5	0.07 ± 0.01 (80.00)		
7	0.02 ± 0.02 (94.28)		
10	N.D.		
	SOIL		
0	1.06 ± 0.07	Y = 3.34 - 0.30X	1.01
1	0.79 ± 0.01 (25.47)		
3	0.47 ± 0.01 (55.66)		
5	0.11 ± 0.00 (89.62)		
7	0.01 ± 0.00 (99.06)		
10	N.D.		

*Mean of 5 replications; N.D. = Not detectable
Figures in parenthesis indicate progressive reduction (%).

Table 2. Effects of decontamination processes on fluvalinate residues in/on chili fruits

Days after treatment	Fluvalinate residues in fresh sample (ppm)*	Residues (ppm)* after Washing	Cooking
0	0.35	0.21 ± 0.01 (40.00)	0.19 ± 0.00 (45.71)
1	0.28	0.13 ± 0.02 (53.57)	0.10 ± 0.02 (64.28)
3	0.13	0.07 ± 0.00 (46.15)	0.06 ± 0.01 (53.84)

*Mean of 5 replications;
Figures in parenthesis indicate progressive reduction (%).

Reports on fluvalinate behaviour on other crops are available (Agnihotri et. al. 1986; Rai et. al. 1986; Gopal et. al. 1987; Mukherjee and

Gopal 1990; Mukherjee et. al. 1992; Agnihotri et. al. 1992). A post application waiting period before harvest of 1 d is suggested for some vegetables after treatment with fluvalinate (Agnihotri et. al. 1992). The persistence of fluvalinate residues on cotton crops and the translocation of its residues into cotton seeds under North Indian climatic condition was reported (Agnihotri et. al. 1986). The half-lives on cotton leaves were found to be 3 d at 50 g a.i./ha ,residues on lint varied from 0.20-1.15 ppm when fluvalinate was applied at 50-100 g a.i./ha. The residues in cotton seeds were un-detectable when applied at 50 g a.i./ha and below tolerence level of 0.20 ppm at 100 g a.i./ha.

The tolerence level of fluvalinate on chili has yet to be established, the residue data generated may help in proposing the MRL of fluvalinate.

Washing and cooking the treated chili fruits quickened the degra - dation of fluvalinate, making them safe for human consumption(Table 2). The same observation was also found in cowpea (Verma and Dixit 1990). In the present investigation, the decontamination processes reduced residues 40-64%. Cooking was more effective than washing in reducing insecticide residues. It may be due to temperature that degrades deposits and translocated portions of insecticide which entered inside the fruits. Translocation of fluvalinate into grains is already reported (Srivastava et. al. 1987). Incedentally, inil deposit in soil was more; although fluvalinate was used as a foliar spray. But it came to a non-detectable level within 10 d of application, without posing soil contamination to a greater extent. The higher initial deposit of fluvalinate in soil might be due to rain (2 mm) that occurred immediately an hour after spraying.

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