Bull. Environ. Contam. Toxicol. (2004) 73:713–716
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DOI: 10.1007/s00128-004-0484-z

Bulletin of Environmental Contamination and Toxicology

Residual Fate of Acrinathrin in Tea Under East-Indian Climatic Condition

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Received: 9 April 2004/Accepted: 14 July 2004

India continues to be the leading producer of tea accounting for about 30% of the global output. The north east (N. E.) India alone accounts for about 75% of total production. Here tea plants are attacked by more than 100 species of pest of which most important one is mite. Various new molecules are extensively used around the world in the control of plant feeding mites. Acrinathrin {(S)-Cyano (3-phenoxyphenyl) methyl [1R-[1a, 3a (Z)]] – 2, 2-dimethyl-3-[3-oxo-3-[2, 2, 2-trifluoro-1-(trifluromethyl)ethoxy]-1-propenyl]cyclopropane}, a novel miticide for crop protection was discovered by Agrochemicals and biological research groups of ROUSSEL UCLAF, belonging to the new chemicals group of non pyrethric esters. It showed a low mammalian toxicity. It acts by contact and ingestion and showed good acaricidal properties against Tetranychidae (Heller *et al.* 1992). Here we carried out a study to determine the dissipation pattern of acrinathrin on made tea and calculate safe waiting period (PHI) for consumption.

MATERIALS AND METHODS

The experiment was laid out in a randomised block design replicated thrice, at Hilla Tea Estate, Jalpaiguri, West Bengal, India during 1995-96 [April 1995 – 1st season, August 1995 – 2nd season, April 1996 - 3rd season] using TV-23 variety. A plot of 100 m^2 was taken for individual treatment. Acrinathrin (Rufast 15 EC) was applied to tea bushes, at the recommended doses 15 g.a.i./ha (T_1) and double the recommended doses 30 g.a.i./ha (T_2) along with untreated control (T_3). The volume of water was 400 l/ha. Tea leaves were plucked randomly from each treatment replication wise at different time intervals [0 (4 hours), 1, 3, 7 and 10 days] after application of the chemical. The green leaf samples were then processed following standard manufacturing method to made tea (CTC, 100 g) in the factory of the Tea estate. Tea samples (25 g) were taken in a conical flask and added 150 ml of acetonitrile and kept for overnight in a dark place. The material was then blended with 20 g of sodium chloride for 5 min. in a Remi automix blender. The extract was filtered through a Buchner funnel. The blender was rinsed twice with 25 ml of acetonitrile and filtered. The combined filtrate was concentrated to 5-10 ml in a rotary vacuum evaporator at 40^0C. The extract was taken in a 500 ml separatory funnel and diluted with 50 ml of saturated aqueous sodium chloride solution and then partitioned with 2 x 50 ml of ethyl acetate and

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Table 1. Persistence of acrinathrin in made tea in Season-I.

Season	Days after application	Treatment	Residue in ppm (M* ± S.D)	Dissipation (%)
	0		3.21±0.60	-
	1	T_1	1.02±0.23	68.22
	3	(15 g.a.i./ha)	0.45±0.16	85.98
Premonsoon	7		0.03±0.16	99.07
(1995)	10		ND	
	0		5.87±0.46	-
	1		3.14±0.23	46.51
	3	T_2	1.25±0.35	78.71
	7	(30 g.a.i./ha)	0.52±0.08	91.14
	10		0.09±0.02	98.47

Regression equation : T_1, Y =3.425-0.277x; T_2, Y =3.71-0.167x
Half life : T_1, 1.09 d; T_2, 1.80 d
T_{MRL} : T_1, 2.08 d; T_2, 1.74 d

Table 2. Persistence of acrinathrin in made tea in Season-II.

Season	Days after application	Treatment	Residue in ppm (M* ± S.D)	Dissipation (%)
	0		2.00±0.47	-
	1	T_1	1.30±0.07	35.00
	3	(15 g.a.i./ha)	0.15±0.06	92.50
Monsoon	7		0.02±0.01	99.00
(1995)	10		ND	
	0		4.46±0.72	-
	1		2.50±0.31	43.95
	3	T_2	2.50±0.31	91.70
	7	(30 g.a.i./ha)	0.37±0.09	98.65
	10		ND	

Regression equation : T_1, Y =3.281-0.294x; T_2, Y =3.594-0.271x
Half life : T_1, 1.02 d; T_2, 1.11d
T_{MRL} : T_1, 2.45 d; T_2, 1.50 d

Table 3. Persistence of acrinathrin in made tea in Season-III.

Season	Days after application	Treatment	Residue in ppm (M* ± S.D)	Dissipation (%)
Premonsoon (1996)	0	T_1 (15 g.a.i./ha)	3.52±0.35	-
	1		1.51±0.31	57.10
	3		0.61±0.07	82.67
	7		0.17±0.05	95.17
	10		0.04±0.03	98.86
	0	T_2 (30 g.a.i./ha)	6.82±0.69	-
	1		3.07±0.07	54.99
	3		1.75±0.29	74.34
	7		0.69±0.18	89.88
	10		0.12±0.06	98.24
Regression equation	:	T_1, Y = 3.431- 0.182x; T_2, Y = 3.756-0.157x		
Half life	:	T_1, 1.65 d; T_2, 1.92 d		
T_{MRL}	:	T_1, 3.13 d; T_2,1.55 d		

M* = Mean of three replications
ND = Not detectable (<0.01 ppm)

organic phase was then dried over anhydrous sodium sulphate and subsequently evaporated to dryness in a rotary vacuum evaporator at 40^0C and the residue was dissolved in 5-10 ml of hexane, which was then subjected to column chromatography (silica gel 60-120 mesh, Qualigen, 25 g) and eluated with 150 ml of hexane : diethyl ether (9:1, v/v). The eluate was then concentrated and volume was made with hexane to 10 ml for analysis. The analysis of acrinathrin residue in made tea was done by HP model 5890A Gas chromatograph with Ni^{63} Electron Capture Detector coupled with 3392A integrator. The glass column (1.8m x 2 mm i.d.) packed with 3% DC-200 on chromosorb WHP 80-100 mesh was used. The oven, injector and detector temperatures were 240, 295 and 275^0C respectively. Flow rate of nitrogen was 60 ml/min. The retention time and minimum detectable limit were 5.04 min and 0.01 μg/g respectively. The average recovery of acrinathrin spiked at 1, 0.5 and 0.1 ppm was 95.80%.

RESULTS AND DISCUSSION

The residue data at different time interval, regression equation, half-life and PHI for acrinathrin were presented in Table 1 to 3. The initial deposits of acrinathrin varied from 2.00 to 3.52 ppm at recommended doses and 4.46-6.82 ppm at double the recommended doses irrespective of any season. It is evident from the Table 1-3, acrinathrin gradually dissipated with increment of time irrespective of any season. In case of recommended dose more than 95% residues dissipated within 7 days and more than 98% in 3 days in monsoon season (August '95). This discrepancy might happen due to heavy down pour during this period. The half-life values were varied from 1.02 to 1.92 days. No residue was found in untreated control (T_3). The MRL value of acrinathrin has not yet been established in Tea. There is no recommended MRL value of acrinathrin in Tea by WHO/FAO. But MRL value of number of synthetic pyrethroids has been fixed in the ranges 10-20 ppm by WHO/FAO. However, Japan regulatory authority has fixed MRL value of acrinathrin as 10 ppm for Tea (Green, Black Oolong, Wulung) (Acrinathrin[103833-18-7] Index from FAN Network Project). From these findings it revealed that residue level of acrinathrin in tea is well below the MRL value. Therefore, we may conclude that acrinathrin may be used safely and there is no necessity to discard first plucking when acrinathrin is applied at the recommended dose.

Acknowledgments . We are very much grateful to M/s. AgrEvo India Ltd. for sponsoring this research programme and Manager, Tea Estate Jalpaiguri, West Bengal for conducting the field experiment.

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