

Persistence of Profenofos Residue on Tea under Northeast Indian Climatic Conditions

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Tea (*Camellia sinensis*) is an important cash crop, earns a lot of foreign exchange through export. Tea is being cultivated mainly in north-east and south India. India is the highest producer of tea in the world. The north-east region of India is producing more than 75% of total production. Indian tea in recent past is facing lot of tough competition with Kenya, Sri Lanka and Bangladesh in respect of tea export as well as in controlling of red spider mites. Besides the above constraints, the importing countries mainly Germany and others European countries are fixing stringent maximum residue limit (MRL) values in respect of pesticide residue occurring in tea. Red spider mite is one of the major pest in tea cultivation specially in north-east region of India. There are a number of miticides are being available, out of which ethion and dicofol are causing residual problem in made tea especially in export. In recent years Profenofos [0-(4-bromo-2-chlorophenyl)-0-ethyl -s-n-propylphosphorothioate], an organophosphorus class of insecticide is used for the control of bollworms, sucking pests like aphids, thrips, jassids and mites on cotton, maize, sugarbeet, soyabean, potatoes, tobacco and other crops (Rengasamy and Dureja 2000, Dhawan and Simwat 1994, Aheer *et al.* 1992, Venugopala Rao *et al.* 1990). In recent years a no. of field trials were reported in various crops viz. tomatoes, aubergines, cucumbers, apples, grapes, strawberry, garlic etc. (Abbassy 2001, Ahmed and Moursy 1991). Based on the above information, a three season field study of Profenofos on tea bushes was conducted in North-East India for determination of the dissipation pattern as well as the residue level of profenofos present in made tea, green tea leaf and tea liquor.

MATERIALS AND METHODS

A three seasons [1st season; October-November 2001 (Post monsoon), 2nd season; April 2002 (pre monsoon), 3rd season; August 2002 (monsoon)], field experiment on tea (variety TV9, TV19) was conducted at Kamalpur Tea estate, Darjeeling, West Bengal during 2001-2002. The commercial formulation of Profenofos (Curacron 50 EC) was applied to tea bushes thrice at an interval of 7 days as high volume spray (400 L/ha) by a Knapsack sprayer. It was applied @ 500 g. a.i./ha (recommended dose i.e. T₁) and 1000 g. a.i./ha (double the recommended dose i.e. T₂) and untreated control (T₃) was simultaneously maintained. Each treatment

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including control was replicated three times in a randomly block designed (RBD). The no. of bushes per treatment were 100 and spacing between the bushes were double hedge type. Green tea leaves (Two leaf and a bud, 1 kg) were plucked randomly from each treatment replicationwise at different time intervals [0 (4 hrs.), 1, 3, 5 and 10 days] after application of Profenofos and the green tea leaf samples processed to made tea (CTC, 100 g) at Kamalpur Tea Estate factory following standard manufacturing methods.

Profenofos (Curacron 50 EC) was extracted from made tea (20 g) with 100 mL methanol in a Remi automix blender (3 min). The homogenate was filtered through a buchner funnel and the residue was re-extracted twice (2 x 30 mL) with methanol and filtered. Then the combined filtrate was transferred in a 500 mL separatory funnel with addition of 100 mL water and 20 mL of saturated sodium chloride solution and then it was partitioned thrice with 100 mL dichloromethane and allowed to separate the phases. The dichloromethane layer was passed through the anhydrous sodium sulphate and combined all the dichloromethane fractions. The combined fraction was concentrated in a rotary vacuum evaporator at 40°C and transferred to a conical flask with hexane. The concentrated extract was chromatographed over a column packed with a silica gel (10g) with 1 cm layer of sodium sulphate at the top. The column was eluted first with mixture of hexane and ether (95:5) and discarded. It was further eluted with 150 mL of mixture of hexane and acetone (95:5). This eluate was then concentrated and volume was made upto 10 mL with acetone for GC-analysis. The same procedure was followed in case of green tea leaf samples.

Made tea (5 g) was brewed with boiled water (100 mL) for 3-5 min. The liquor was filtered and cooled and then partitioned with (3 x 50 mL) dichloromethane. The combined extract was concentrated to a small volume and dissolved in hexane. Similar steps as described for made tea were followed for determining the residue in tea liquor.

Final analysis of Profenofos (Curacron 50 EC) residue in made tea, green tea leaf and tea liquor samples were done by gas chromatograph (H. P. model 5890 series II) with flame photometric detector coupled with 3392A, integrator. The glass column (6' x 0.2 mm i.d.) packed with 3% OV-101 on chormosorb WHP 60-100 mesh was used. The temperature were : Oven 220°C, Injector 240°C, Detector 250°C. Flow rate of carrier gas (nitrogen), hydrogen and air were 50, 30 and 60 ml/min respectively. The retention time, sensitivity and limit of detection were 1.12 min, 0.005 µg/g and 0.01 µg/g respectively.

The average recovery of Profenofos in made tea, green tea leaf and tea liquor spiked at 0.5, 1 and 5 ppm were 89, 85.5 and 87.8% respectively.

RESULTS AND DISCUSSION

The residue data of Profenofos (Curacron 50 EC) and regression equation, half-life for consecutive three seasons at different days interval are represented in the

tables (1-9). Interestingly, the result showed that the residues of Profenofos in made tea as well as in green tea leaves declined progressively with time irrespective of any dose and season. The initial deposit (4 hr. after spraying) of Profenofos were found to be in the range of 15.48-21.25 ppm irrespective of any season at the recommended dose (T_1) and 33.46-35.68 ppm at double the recommended dose (T_2). No residue was detected in the untreated control (T_3) samples. It was observed from this study that dissipation rate was very fast and 50% residue dissipated within 0.79 - 1.20 days in respect of made tea whereas in case of green tea leaf it ranged from 0.77-1.01 days irrespective of any season and dose. In made tea and green tea leaf, about 70% Profenofos residue dissipated within 3 days after last application irrespective of any dose and season (Table 1-6). No residue was detected in made tea as well as green tea leaf samples on the 5th day after application at the recommended dose, i.e., 500 g.a.i./ha irrespective of any season. It is evident from tables (7-9) that more than 90% residue of Profenofos was diminished in tea liquor on and from 3rd day after last application. No residue was detected in the tea liquor in 5th day samples irrespective of any dose and season. The MRL value of Profenofos has not yet been established in tea. There is no recommended MRL value of Profenofos in Tea by WHO/FAO. But MRL value of Profenofos in different substances has been fixed in the range of 0.05-2 ppm by WHO/FAO. As no residue was detected in the 5th day sample it might be stated that Profenofos may not pose any residual toxicity problem in tea which is also befitting with the plucking schedule of Tea Estates of North-East region of our country.

Table 1. Persistence of profenofos in made tea in season-I.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Post Monsoon (2001)	0		21.25±9.26	-
	1		8.72±1.58	58.97
	3	T_1	3.55±0.91	59.28
	5	(500 g.a.i./ha)	ND	-
	10		ND	-
	0		33.46±7.28	-
	1		10.46±2.82	68.73
	3	T_2	6.85±1.80	34.51
	5	(1000 g.a.i./ha)	0.34±0.15	95.03
	10		ND	-
Regression equation		: T_1 , $Y = 4.274 - 0.250x$; T_2 , $Y = 4.540 - 0.362x$		
Half life		: T_1 , 1.20 d; T_2 , 1.84 d		

Table 2. Persistence of profenofos in made tea in season-II.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Pre Monsoon (2002)	0		18.45±4.23	-
	1	T ₁	8.91±1.88	51.71
	3	(500 g.a.i./ha)	2.84±6.98	84.61
	5		ND	-
	10		ND	-
	0		35.03±4.91	-
	1	T ₂	14.73±2.45	57.95
	3	(1000 g.a.i./ha)	7.37±0.84	78.96
	5		0.43±0.10	98.77
	10		ND	-
Regression equation			: T ₁ , Y = 4.242 – 0.268x; T ₂ , Y = 4.608-0.359x	
Half life			: T ₁ , 1.12 d; T ₂ , 0.89 d	

Table 3. Persistence of profenofos in made tea in season-III.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Monsoon (2002)	0		15.48±2.91	-
	1	T ₁	8.04±0.95	48.06
	3	(500 g.a.i./ha)	2.22±0.60	85.66
	5		ND	-
	10		ND	-
	0		35.68±6.41	-
	1	T ₂	13.93±3.70	60.96
	3	(1000 g.a.i./ha)	6.75±1.11	81.08
	5		0.41±0.10	98.85
	10		ND	-
Regression equation			: T ₁ , Y = 4.187 – 0.382x; T ₂ , Y = 4.592-0.360x	
Half life			: T ₁ , 0.79 d; T ₂ , 0.84 d	

Table 4. Persistence of profenofos in/on green tea leaf in season-I.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Post Monsoon (2001)	0		2.63±0.62	-
	1	T ₁	0.83±0.24	68.44
	3	(500 g.a.i./ha)	0.17±0.09	79.51
	5		ND	-
	10		ND	-
	0		5.53±1.45	-
	1	T ₂	2.47±1.45	55.33
	3	(1000 g.a.i./ha)	0.69±0.12	72.06
	5		0.17±0.08	75.36
	10		ND	-
Regression equation			: T ₁ , Y = 3.376 – 0.389x; T ₂ , Y = 3.719-0.297x	
Half life			: T ₁ , 0.77 d; T ₂ , 1.01 d	

Table 5. Persistence of profenofos in/on green tea leaf in season-II.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Pre Monsoon (2002)	0		3.44±0.87	-
	1	T ₁	1.22±0.26	64.53
	3	(500 g.a.i./ha)	0.27±0.09	77.86
	5		ND	-
	10		ND	-
	0		7.07±1.30	-
	1	T ₂	2.89±0.65	59.12
	3	(1000 g.a.i./ha)	0.76±0.22	73.70
	5		0.17±0.06	77.63
	10		ND	-
Regression equation			: T ₁ , Y = 3.506 – 0.364x; T ₂ , Y = 3.821-0.318x	
Half life			: T ₁ , 0.83 d; T ₂ , 0.95 d	

Table 6. Persistence of profenofos in/on green tea leaf in season-III.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Monsoon (2002)	0	T ₁ (500 g.a.i./ha)	3.70±0.69	-
	1		1.26±0.29	65.94
	3		0.25±0.12	80.15
	5		ND	-
	10		ND	-
	0	T ₂ (1000 g.a.i./ha)	7.23±0.83	-
	1		2.66±0.67	63.20
	3		0.82±0.24	69.17
	5		0.18±0.04	78.04
	10		ND	-
Regression equation		: T ₁ , Y = 3.530 – 0.380x; T ₂ , Y = 3.808-0.393x		
Half life		: T ₁ , 0.79 d; T ₂ , 0.77 d		

Table 7. Persistence of profenofos in tea liquor in season-I.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Post Monsoon (2001)	3	T ₁ (500 g.a.i./ha)	0.12±0.06	-
	5		ND	-
	10		ND	-
	3	T ₂ (1000 g.a.i./ha)	0.17±0.05	-
	5		ND	-
	10		ND	-

Table 8 : Persistence of profenofos in tea liquor in season-II.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Pre Monsoon (2002)	3	T ₁ (500 g.a.i./ha)	0.08±0.03	-
	5		ND	-
	10		ND	-
	3	T ₂ (1000 g.a.i./ha)	0.18±0.03	-
	5		ND	-
	10		ND	-

Table 9. Persistence of profenofos in tea liquor in season-III.

Season	Days after application	Treatment	Residue in ppm (M*±S.D)	Dissipation (%)
Monsoon (2002)	3	T ₁ (500 g.a.i./ha)	0.16±0.05	-
	5		ND	-
	10		ND	-
	3	T ₂ (1000 g.a.i./ha)	0.27±0.09	-
	5		ND	-
	10		ND	-

M* = Mean of three replications

ND = Not detectable (<0.01 ppm)

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