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Residues of Amitraz, a New Acaricide, on Tea

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India is one of the major tea producing country and earns a lot of foreign exchange by exporting tea. In recent years it has been observed that tea export is declining due to several reasons. Our country is facing tough competition with Kenya and Sri Lanka in respect of tea export. Today the global tea market has centralised in Germany and other European countries and considering health point of view they are gradually lowering their maximum residue limit (MRL) of different pesticides in all food items including tea.

In recent years red spider mite is one of the key pests in tea plantation. After the banning of tetradifon and restricting the use of dicofol and ethion a vacuum is created with respect of acaricides. Very recently a number of acaricides are going to be introduced in India. Amongst the new acaricides, Amitraz (Mitac 20EC) [N-methyl bis (2, 4-xylylimino methyl amine)] and Fenazaquin would be probable choices. Very little information is available on the efficacy of amitraz against red spider mite on tea. It is widely used for the control of *Polyphagotarsenomus latus*, *Tetranychus ludeni, T. urticae, T. cinnabarinus* on cotton, chilli, pea, bean, apple in different parts of the world (Sannaveerappanavar and Channa Basavanna 1986; Rajasri *et al.* 1991; Cheng and Pan 1994). Recently bioefficacy of amitraz has been evaluated by our entomologists against plant feeding mites occuring in tea and from the results it appears to be the most effective one (Saha 1998). Considering the above facts, a three season field study on tea bushes was conducted in Dooars' area for the determination of the dissipation pattern as well as the residue level of amitraz present in made tea.

MATERIALS AND METHODS

The experiment was conducted at Debpara (pre-monsoon, 1995) and Hilla Tea Estate (monsoon, 1995 and pre-monsoon, 1996). Variety, TV9, TV19 (mixed clone); plot-size, 100 sq. m.; spacing, 100 cm x 60 cm x 60 cm,

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double hedge type; design, RBD.

Amitraz was applied to tea bushes at the recommended rate 200 g a.i./ha (T_1) double the recommended rate 400 g a.i./ha (T_2) alongwith untreated control (T_3). Replication, three; total volume of water, 400 L/ha. After plucking the tea shoots, amitraz was applied with Knapsac sprayer.

Tea leaves (two leaf and a bud) were plucked at different days [0 (4 hr), 1, 5, 10 and 14 d] after application of the insecticide. The green leaf samples were then processed by standard techniques in the factory and made tea (CTC) samples were collected.

Made tea (20g) sample was extracted with 150 mL of 2 *M* hydrochloric acid for 18 hr in a reflux condition. The extract was allowed to cool at room temperature and filtered through a Buchner funnel. Then the filtrate was neutralised with 25 mL of 14 *M* sodium hydroxide and the basic portion was partitioned thrice with *n*-hexane (100 + 50 + 50) mL. After discarding the aqueous portion the hexane fraction was concentrated in a rotary vacuum evaporator. The concentrated mass in hexane was then partitioned twice with 2 *M* hydrochloric acid (2 + 1) mL and discarded the hexane fraction and basified with 10 *M* sodium hydroxide (1mL), subsequently partitioned with the 2x5 mL of *n*-hexane. Aqueous layer was discarded. The combined hexane fraction was passed through anhydrous sodium sulphate and collected in a test tube. 50 µL heptafluorobutyric anhydride was added to the hexane fraction in cold condition. The test tube was then kept on water bath at 50°C for 20 min. The mixture was cooled at room temperature and then were added 1 mL of distilled water and 1 mL of 3 *M* ammonia solution. Hexane layer was transfered to a 10 mL volumetric flask and made up to the mark with hexane, ready for GC analysis.

Final analysis of amitraz as derivative of 2, 4 - dimethylaniline in made tea was done with HP Model 5890A Gas Chromatograph with ECD (Ni^{63}) coupled with 3392A integrator. The GC conditions were as follows :

Column, DB-5 [length 30 m, id 0.53 mm (Megabore), film thickness 1.5 µ (J & W Scientific)]; oven temperature (initial), 100°C; oven temperature (final), 180°C; injector temperature, 210°C; detector temperature, 280°C; initial time, 2 min; final time, 10 min; rate, 10°C/min; flow rate of carrier gas (N_2), 30 mL/min; retention time, 4.47 min; limit of detection, 0.01 ppm.

A stock solution of 0.5–1 ppm analytical grade 2, 4 - dimethylamiline (99% purity) was derivatised with heptafluorobutyric anhydride following the above method and this derivative was used as an external standard.

RESULTS AND DISCUSSION

The average recovery of amitraz following the analytical procedure was found to 80 to 92% from samples fortified with 0.5, 1 and 5 ppm of analytical standard. The residue data of amitraz at different d interval in three different seasons were presented in Table 1, 2 and 3.

Application of amitraz at 200 g a.i./ha and 400 g a.i./ha was found to leave 6.20 and 10.35 ppm in 1995 trial (season I), 4.03 and 6.24 ppm in 1995 trial (season II) and 5.58 and 12.65 ppm in 1996 trial (season III) respectively on made tea. The wide variation in the initial deposits could be attributed due to influence of weather.

In rainy season (1995) at 0 d the rainfall occured 6 hr after application. It rained almost every day except 8^{th} – 11^{th} d. This may lead to poor deposition. After 10 d the residues were remained 0.22 and 0.43 ppm for T_1 and T_2 respectively. In 1995 trial (season I) residues were found after 5 d 0.95 and 2.03 ppm for T_1 and T_2 respectively. The dissipation percent in 1996 trial (season III) after 5 d were 73.11 and 79.44 for T_1 and T_2 respectively. No residue was detected in untreated control (T_3).

In all the trials more than 90% of the applied pesticide was lost within 10 d after application. The amount of rainfall was higher in season II than the other two seasons. The dissipation followed first order reaction kinetics irrespective of any treatments and seasons. A straight line was obtained in each case when log residues were plotted against different time interval (Fig 1, 2 and 3) and the half-life values were calculated and it ranges from 1.97 to 2.92 d.

From this study it is observed that amitraz is dissipated quickly with the increment of time. After 10th d the residue persisted in the range of 0.15-0.27 ppm when applied at the recommended dose. Interestingly, MRL value of amitraz on made tea is not yet fixed. Considering the residue of amitraz present at 10th d coupled with plucking schedule (6-7 d interval) it might be stated that amitraz will not pose any residual toxicity problem in Tea.

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Table 1. Persistence of amitraz in made tea in different seasons (season I).

Season	Days after application	Treatment	Residue in ppm (M* ± S.D.)	Dissipation (%)
	0		6.20 ± 0.496	–
	1		2.34 ± 0.237	62.25
	5	T_1	0.95 ± 0.120	84.67
	10	(200 g. a.i./ha)	0.15 ± 0.033	97.58
Pre-monsoon	14		ND	
(1995)				
	0		10.35 ± 0.984	–
	1		6.80 ± 0.427	34.29
	5	T_2	2.03 ± 0.094	80.38
	10	(400 g. a.i./ha)	0.45 ± 0.086	95.65
	14		0.06 ± 0.022	99.42
		T_3	ND	
Regression equation :		T_1, Y = 2.67 – 0.149 X ; T_2, Y = 3.03 – 0.153 X		
Half-life :		T_1, 2.02 d ; T_2, 1.97 d.		

Table 2. Persistence of amitraz in made tea in different seasons (season II).

Season	Days after application	Treatment	Residue in ppm (M* ± S.D.)	Dissipation (%)
	0		4.03 ± 0.46	–
	1		0.85 ± 0.085	78.90
	5	T_1	0.58 ± 0.028	85.60
	10	(200 g. a.i./ha)	0.22 ± 0.036	94.54
Monsoon	14		ND	
(1995)				
	0		6.24 ± 0.343	–
	1		1.83 ± 0.432	70.67
	5	T_2	1.03 ± 0.15	83.49
	10	(400 g. a.i./ha)	0.43 ± 0.028	93.10
	14		0.11 ± 0.022	98.23
		T_3	ND	
Regression equation :		T_1, Y = 2.32 – 0.103 X ; T_2, Y = 2.59 – 0.107 X		
Half-life :		T_1, 2.92 d ; T_2, 2.81 d.		

Table 3. Persistence of amitraz in made tea in different seasons (season III).

Season	Days after application	Treatment	Residue in ppm (M* ± S.D.)	Dissipation (%)
Pre-monsoon (1996)	0	T_1 (200 g. a.i./ha)	5.58 ± 0.235	–
	1		2.70 ± 0.297	51.61
	5		1.50 ± 0.214	73.11
	10		0.22 ± 0.045	96.05
	14		ND	
	0	T_2 (400 g. a.i./ha)	12.65 ± 1.043	–
	1		5.50 ± 727	56.52
	5		2.60 ± 0.412	79.44
	10		0.39 ± 0.050	96.91
	14		0.08 ± 0.016	99.36
		T_3	ND	
Regression equation :		T_1, Y = 2.69 – 0.130 X ; T_2, Y = 3.04 – 0.149 X		
Half-life :		T_1, 2.32 d ; T_2, 2.02 d.		

ND = not detectable (<0.01 ppm)
* = average of three replications

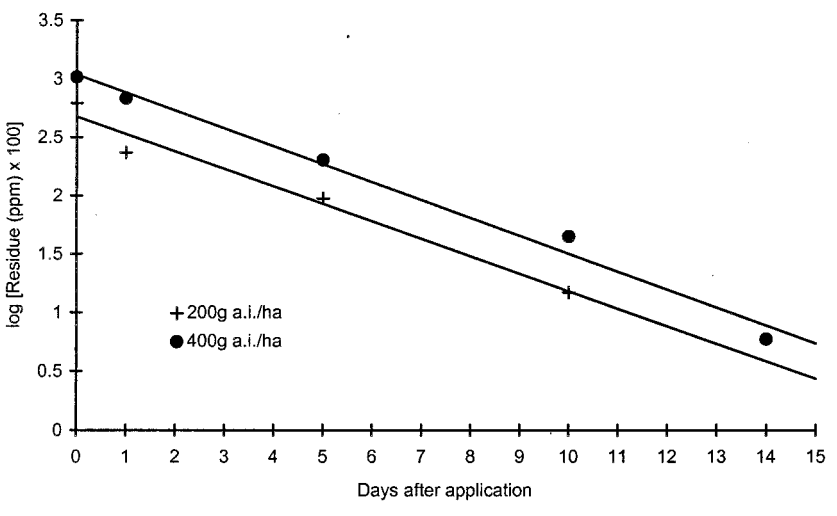

Figure 1. Linear plot for first order reaction kinetics of amitaz in made tea (Pre-monsoon 1995)

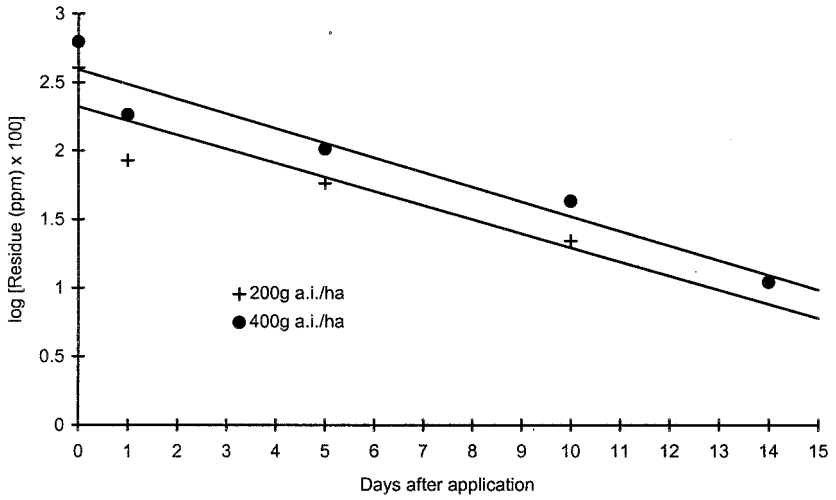

Figure 2. Linear plot for first order reaction kinetics of amitaz in made tea (Monsoon 1995)

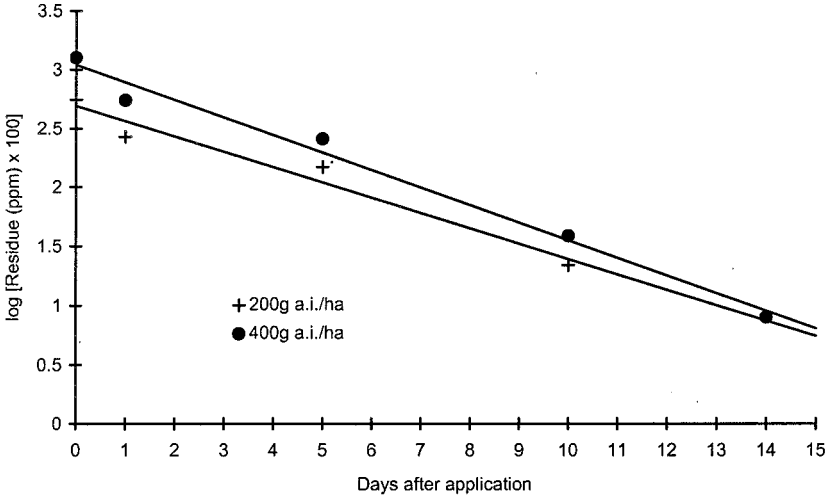

Figure 3. Linear plot for first order reaction kinetics of amitaz in made tea (Pre-monsoon 1996)

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