Bull. Environ. Contam. Toxicol. (2004) 73:347–350

DOI: 10.1007/s00128-004-0434-9

Bulletin of Environmental Contamination and Toxicology

Persistence of Dicofol Residue on Tea Under North-East Indian Climatic Conditions

K. Saha,[1] T. Saha,[2] H. Banerjee,[2] A. Bhattacharyya,[2] A. Chowdhury,[2] A. K. Somchoudhury[1]

[1] Department of Agricultural Entomology, Department of Agricultural Chemicals, Bidhan Chandra Krishi Viswavidyalaya, Mohanpur 741252, West Bengal, India
[2] Pesticide Residue Laboratory, Department of Agricultural Chemicals, Bidhan Chandra Krishi Viswavidyalaya, Mohanpur 741252, West Bengal, India

Received: 24 June 2003/Accepted: 14 May 2004

Tea, *Camellia sinensis* (L.) O. Kuntze, an important cash crop, is cultivated in northeast and South India. The northeast India alone accounts for 75% of total production. Our country is earning good amount of foreign exchange by exporting Tea. It has been observed that more than a hundred species of pest occurred in Tea plants of N. E. India of which the most important one is plant feeding mite, *viz.,* red spider mite, *Oligonycus coffeae* (Nietner), scarlet mite, *Brevipalpus phoenices* (Geijskes) are major and pink mite, *Acaphylla theae* and purple mite, *Calacarus carinatus* are minor in nature N. E. India (Banerjee, 1988). Dicofol, an acaricide, is very effective against red spider mite of tea. It has a good knockdown effect, but acts mostly on the adult stages of mites (Ananthakrishnan,1963; Carnham,1963; Mukherjee,1963andBanerjee,1979). Therefore the present study was carried out in order to determine the persistence nature of dicofol 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, WB, India during 1995-96 (April 1995-1st season, August, 1995-2nd season, April 1996-3rd season) using TV1 variety. A plot of 100 m2 was taken for individual treatment. Dicofol (18.5 EC, Kelthane) was applied to tea bushes at the recommended doses @ 170 g a.i./ha (1:400 dilution T1) and also at double the recommended doses @ 340 g a.i./ha (1:200 dilution, T2) along with untreated control (T3). The volume of water was used 400 1 ha-1. Tea leaves were plucked randomly from each treatment replication wise at different time interval [0 (4 hr), 1,3,7 and 10 days] after application of the chemical. The green leaf samples were then processed in the factory of the Tea garden following standard manufacturing method to made tea (CTC 100g). Tea samples (10 g) were homogenized with 150 ml acetone in a Remi automix blender (3 min). The homogenate was filtered through buchner funnel and residue was re-extracted twice (2 x 25 ml) with acetone and filtered. The combined filtrate was concentrated (50 ml) by a rotary vacuum evaporator at 40^0 C. The concentrated extract was then transferred into a 500 ml separatory funnel with the addition of 50 ml of aqueous saturated sodium chloride solution and 100 ml of n-hexane and

Correspondence to: A. Bhattacharyya

shaken vigorously (2-3 min). The hexane layer was passed through the anhydrous sodium sulphate. Then aqueous layer was extracted twice with 50 ml n-hexane and the upper layer was collected similarly and combined all the hexane fractions. The hexane fractions was concentrated by a rotary vacuum evaporator. The concentrated extract was then subjected to column chromatography over activated florisil (60-100 mesh, Spectrochem, 15 g) with a 12 cm layer of anhydrous sodium sulphate at the top. The column was eluted with a mixture of n-hexane-diethyl ether (9:1, 150ml). The eluate was then concentrated and volume was made up to 10 ml with n-hexane for GLC analysis. The analysis of dicofol residue in made tea was done by H. P. Model 5890A gas Chromatograph with Ni^{63} Electron Capture Detector Coupled with 3392A Integrator. The glass column (1.8 m x 2 mm i.d.) packed with 3% DC-200 on chromosorb WHP 80-100 mesh was used. The temperatures were: column 200^0, injector 220^0 and detector 300^0 C. Flow rate of carrier gas (nitrogen) was 40 ml/min. The retention time and limit of detection were 1.82 min and 0.01 µg/g respectively. The average recovery of dicofol spiked at 1, 0.5 and 0.1 ppm was 93.46%.

RESULTS AND DISCUSSION

Residual data of dicofol (Kelthane 18.5 EC) in three different seasons were represented in table1-3.The corresponding regression equation, half-life, T_{MRL} (waiting period or pre-harvest interval or PHI) have also been calculated on the basis residue data. The initial deposit (4 hr. after spraying) of dicofol were found to be in the range of 4.70 to 22.81 ppm irrespective of any season at the recommended dose (T_1) and 8.71 to 32.81 ppm at double the recommended dose (T_2). No residue was detected in the untreated control (T_3) samples. It is evident from the table (1-3) that the dicofol residue gradually dissipated with increment of

Table 1. Persistence of dicofol in brewed tea in the spring

Season	Days after application	Treatment	Residues in ppm Mean ± SD	Dissipation (%)
Pre-monsoon April,1995	0	T_1 (170 g. a.i./ ha)	19.74 ± 1.02	-
	1		14.37 ± 0.58	27.20
	3		11.24 ± 0.79	41.06
	7		5.39 ± 0.80	72.70
	10		1.08 ± 0.35	94.53
	0	T_2 (340 g.a.i./ha)	27.16 ± 0.56	
	1		21.72 ± 0.93	20.03
	3		17.50 ± 0.50	35.57
	7		10.35 ± 1.07	61.89
	10		3.05 ± 0.13	88.77
Regression equation: T_1 : y = 3.341 - 0.116x; T_2 : y = 3.466 - 0.087x; Half life : T_1 : 2.60 d ; T_2 : 3.46 d; T_{MRL}: T_1 : 5.53 d ; T_2 : 8.82 d				

time. The most salient feature of this study that the level of dicofol residue present on 10th day sample was around 1 ppm at the recommended dose whereas, at double the recommended doses it ranged from 1.74 to 4.36 ppm. The half- life values ($T_{1/2}$) were found to be in the range of 2.60-5.10 irrespective of any doses and seasons.

It is evident from the meteorological data of three different seasons that rainfall has significant contribution towards poor deposition of dicofol in the monsoon

Table 2. Persistence of dicofol in brewed tea in summer

Season	Days after application	Treatment	Residues in ppm Mean ± SD	Dissipation (%)
Monsoon August,1995	0	T_1 (170 g. a.i./ ha)	4.70 ± 0.47	-
	1		3.12 ± 0.50	33.62
	3		2.37 ± 0.26	49.57
	7		1.92 ± 0.16	59.15
	10		0.93 ± 0.14	80.21
	0	T_2 (340 g.a.i./ha)	8.71 ± 0.86	-
	1		5.96 ± 0.59	31.57
	3		4.26 ± 0.67	51.09
	7		3.74 ± 0.19	57.06
	10		1.74 ± 0.47	80.02
Regression equation: T_1 : y = 2.606 - 0.060x; T_2 : y = 2.882 - 0.059x; Half life : T_1 : 5.02 d ; T_2 : 5.10 d; T_{MRL}: T_1 : 1.55 d ; T_2 : 3.10 d				

Table 3. Persistence of dicofol in brewed tea in spring

Season	Days after application	Treatment	Residues in ppm Mean ± SD	Dissipation (%)
Pre-Monsoon April,1996	0	T_1 (170 g.a.i./ ha)	22.81 ± 0.75	-
	1		17.37 ± 0.70	23.85
	3		13.05 ± 0.69	42.79
	7		6.25 ± 0.21	72.60
	10		1.34 ± 0.13	94.13
	0	T_2 (340 g.a.i./ha)	32.81 ± 0.84	-
	1		21.35 ± 0.18	34.93
	3		16.25 ± 0.81	50.47
	7		10.28 ± 0.26	68.67
	10		4.36 ± 0.79	86.71
Regression equation : T_1 : y = 3.406 - 0.113x ; T_2 : y = 3.471 - 0.078x Half life : T_1 : 2.66 d; T_2 : 3.86 d ; T_{MRL} : T_1 : 6.25 d; T_2 : 9.90 d				

(August, 1995). But in the dry season (pre-monsoon) the initial deposit of dicofol was as high as 32.81 ppm. However the dissipation pattern is almost similar and the rate of dissipation followed the first order kinetics irrespective of any dose or season.On the basis of MRL (5 ppm) (Anonymous,1975) the T_{MRL} values for T_1 and T_2 were calculated in the range of 1.55 to 6.25d & 3.10 to 9.90d respectively.

Recently, Barooah *et al.* (1995) determined the PHI values of dicofol in made tea from two different field trails namely Tocklai and Darjeeling. The PHI values were calculated as 4.64 and 4.32 for Tocklai and Darjeeling respectively the results of which are comparable to our study. From waiting period data it might be stated that the one round of plucking may be discarded when dicofol is applied at the recommended doses.

Acknowledgments. We thank M/S Indofil Co. Ltd., Mumbai, India for financial support and to BCKV for infrastructural facilities.

REFERENCES

Anonymous (1975) Joint FAO/WHO report of the eight session of the Codex Committee on pesticide residue. Codex Alimentarius Commission, FAO/WHO, Geneva, pp 45-54.

Ananthakrishnan NR (1963) Report of entomologist, Ann Rept Tea Sci UPASI, pp 40-51.

Banerjee B (1979) Arthropod diversity on a monoculture of tea under shade and unshaded condition. Sci Cult 45 : 454-455.

Banerjee B (1988) An introduction to agricultural acarology, Associated Pub Com. New Delhi p118.

Barooah AK, Kalita JN, Collier PD and Barbora BC (1995) Residues of pesticides in made tea and hot water brew. Two and a bud pp196-214.

Carnham JE (1963) Report of entomologist, Rept Tea Res Inst Ceylon for 1962, pp50-69.

Mukherjee TD (1963) New pesticides. Two and a bud 10 : 32-36.