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Determination of Bifenthrin Residues in Cotton

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Studies were conducted to evaluate the harvest time residues of bifenthrin 10 EC on cotton in Tamil Nadu Agricultural University, Coimbatore. The treatments were bifenthrin 10 EC at 80 g a.i. ha^{-1}, 160 g a.i. ha^{-1} and untreated check. Samples were collected at random on 100 and 115 days after treatment for analysis. The results revealed that the harvest time residues of bifenthrin 10 EC at 80 and 160 g a.i. ha^{-1} were at below detectable level in lint, seed, oil and soil samples.

Key words: Bifenthrin, residues, cotton, pyrethroids

Cotton (*Gossypium* spp) is the most important cash crop and natural textile fibre in India and popularly known as “White gold” or “King of fibre crops”. In India, cotton occupies a prime position with 20 per cent cultivated area and 12 per cent global production but consumes more than 55 per cent of total insecticides used in the country (Puri, 1995). Among the various cardinal factors responsible for the low yield of cotton in India, the losses due to large number of insect pests are of prime importance. A number of insecticides have been tried by different workers to control these pests. Synthetic pyrethroids were readily adopted by farmers in cotton because of their superiority over other insecticides in controlling bollworm complex (Jayswal and Saini, 1981: Agnihotri *et al.,* 1986). Main reason behind the heavy use of pesticides in cotton ecosystem is due to the injudicious use of toxic pesticides and other inputs. Several problems like development of pest resistance to insecticides, pest resurgence and build up of bio concentrations of pesticide residues at harvest were resulted. Further, no uniform adoption of pesticides in different regions, cropping system and public health programmes which again deepen the above consequences. Hence keeping this in view, the present study was undertaken to determine the harvest time residues of bifenthrin 10 EC in cotton lint, seed, oil and soil.

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Materials and Methods

The study was undertaken to find out the levels of residues of bifenthrin in cotton lint, seed, oil and cropped soil. The field trial was laid out at Mandapasalai, near Aruppukottai, Tamil Nadu, in a randomized block design with a plot size of $40m^2$ with three replications using the variety Ankur WH-216. The treatments are bifenthrin 10 EC @ 80 g a.i. ha^{-1} (T_1) and bifenthrin 10 EC 160 g a.i. ha^{-1} (T_2) along with untreated check (T_3). Treatments were imposed three times at an interval of 15 days using a high volume sprayer. Sampling of cotton kapas was done during the first and second pickings. Samples were ginned to analyse residues in the seed, lint and oil.

Extraction and Clean up

Lint

The representative lint sample (5 g) was soaked in 100 ml of acetone overnight for extraction. The contents were filtered through a Buckner funnel and the residue was extracted with 2 x 50 ml of n – hexane : acetone mixture (1:1) and again filtered. The combined n – hexane : acetone mixture was evaporated in a rotary vaccum evaporator to 10 – 15 ml. Then it was passed through sodium sulphate and condensed in a rotary vaccum evaporator to 1 – 2 ml. Finally it was transferred to a graduated tube and made upto 10 ml using n – hexane : acetone mixture (1:1) for GC analysis.

Seed

Ten gram of seed was ground with little quantity of anhydrous sodium sulphate and soaked in 100 ml of acetone overnight. The contents were filtered through a Buckner funnel and the residue was extracted with 2 x 50 ml of n – hexane : acetone mixture (1:1) and again filtered. The combined n – hexane : acetone mixture was evaporated in rotary vaccum evaporator. The concentrated solution was transferred in a separating funnel and then 100 ml of saturated sodium chloride solution was added, followed by 100 ml of n – hexane. The mixture was shaken vigorously and allowed to stand for phase separation. The upper organic phase (hexane layer) was separated and the aqueous layer was extracted twice with 50 ml of n – hexane. The combined n – hexane layer was dried by passing it through anhydrous sodium sulphate. The dried n – hexane phase was concentrated to 10 ml by heating it in waterbath at 35^0 C. The residual solution was then subjected to florisil column clean up. The chromatographic column was plugged with cotton wool and 2 g of anhydrous sodium sulphate was first added and then deactivated florisil 5 g was slowly added to the column and again 2 g of anhydrous sodium sulphate was added on the top of the column.

After pre-washing with hexane, 10 ml of concentrated extract was transferred to the column. The beaker was rinsed with 2 x 5 ml of n – hexane and this was added to the column. Then 50 ml of n – hexane was added to the column, allowed to elute and discarded. The bifenthrin was eluted from the column with 150 ml of (5% v/v) ethyl acetate in hexane, the elutant was collected in a flask, concentrated in a rotary vaccum evaporator and the volume was finally made upto 10 ml with n – hexane : acetone mixture (1:1) for analysis by GC.

Oil

Oil was extracted from the seeds with acetone in Soxhlet apparatus. Then the same procedure as described for seed was followed for extraction and clean up of oil.

Soil

Fifteen gram of soil sample was taken by following quartering technique and mixed with 2g of florisil, 5g of anhydrous sodium sulphate and a pinch of activated charcoal. The chromatographic column was plugged with cotton wool and 2g of anhydrous sodium sulphate was first added and then the prepared soil mixture was packed, followed by another 2g of anhydrous sodium sulphate on the top of the column. The remaining clean up procedure was followed as described for seed.

Final determination

Gas chromatography (GC Chemito 2865) equipped with electron capure detector (Ni^{63}) was used for final determination of the bifenthrin residues with the following operating parameters

Column	: 4 % S.E. 30 on Anachrome Q
Oven temperature	: 220^0C
Injection temperature	: 260^0C
Detector temperature	: 280^0C
Gas flow (N_2)	: 20 ml min^{-1}
Retention time	: 4.17 min.
Limit of detection	: 0.001 ppm

Preparation of standards and recovery studies

Bifenthrin technical material was obtained from M/s. United Phosphorus Ltd., Mumbai. The stock solution of 1000 ppm was prepared by dissolving 100 mg of technical material (99.7 per cent purity) in 100 ml of HPLC grade acetone. From this stock, intermediate stock solutions of 100 ppm and 10 ppm were prepared. Using 10 ppm stock, working standards of 0.5, 1 and 2 ppm were prepared in acetone. Samples were fortified with working standards to find out the recovery of bifenthrin. The recovery factor worked out was taken for final calculation.

Calculation

Bifenthrin standards were used for recording the retention time and it was 4.17 minutes. Then the samples were subjected for injection in HPLC. Bifenthrin residue was identified by comparing the retention time of sample peaks with that of the standard and the amount of residue was

recorded in the chart. The amount of residue in ppm (µg g^{-1}) was calculated as follows.

$$\text{Residues (in ppm)} = \frac{A_1 \times C \times V_1}{A_2 \times W \times V_2} \times RF$$

Where

A_1 = Area of sample

A_2 = Area of standard

C = Concentration of standard in ppm

RF = Recovery factor

V_1 = Total volume of sample in ml

V_2 = Injected volume of the sample in µl

W = Weight of the sample in gram

Results and Discussion

Recovery studies were carried out in order to establish reliability of the analytical methods and to know the efficiency of extraction and clean up steps employed for the present study by fortifying the lint, seed, oil and soil samples with analytical standard of bifenthrin at 1, 2 and 5 ppm level. The results of the recovery study are presented in Table 1. The mean recovery of bifenthrin was 86.90, 85.03, 85.23 and 89.67 per cent from fortified lint, seed, oil and soil samples, respectively. The minimum detection limit of the instrument was 0.5 ng and the determinability level in lint and soil was 0.1 µg g^{-1} considering 10 g weight of each sample and final volume of the extract was

Table 1. Recovery of bifenthrin from lint, seed, oil and soil samples of cotton

Matrix / Substrate	Amount fortified (ppm)	Amount recovered* (ppm)	Recovery (%)	Average recovery (%)
Lint	1.0	0.88	88.0	
	2.0	1.65	82.5	86.90
	5.0	4.51	90.2	
Seed	1.0	0.82	82.0	
	2.0	1.71	85.5	85.03
	5.0	4.38	87.6	
Oil	1.0	0.85	85.0	
	2.0	1.69	84.5	85.23
	5.0	4.31	86.2	
Cropped soil	1.0	0.90	90.0	
	2.0	1.72	86.0	89.67
	5.0	4.65	93.0	

***** Average of three replicates

2 ml. The determinability level in seed and oil was 0.05 and 0.2 µg g^{-1} considering the weight of the sample 20 g and 5 g, respectively and final volume of the extract was 2-5 ml. The level of residues of bifenthrin 10 EC at 80 and 160 g a.i.ha^{-1} as foliar spray in cotton were at below detectable level in lint, seed and oil samples of first as well as second pickings and soil samples collected from two field experiments (Table 2). The time interval between last spray and first and second pickings / harvest was 100 and 115 days, respectively. Present investigation is in accordance with the findings of Nivedita *et al.* (1998) who reported that bifenthrin residue was not found or below detectable level on 14 days after spray at 20 and 40 g a.i. ha^{-1} concentration in chillies. Bifenthrin was safer in controlling sucking pests of cotton without residual problem.

Table 2. Harvest time residues of bifenthrin in lint, seed, oil of cotton and soil

Harvest Time	Treatment	Dose (g.a.i.ha^{-1})	Replication	Residues in ppm			
				Lint	Seed	Oil	Soil
I	T_1	80	R_1	BDL	BDL	BDL	BDL
(100 DAS)			R_2	BDL	BDL	BDL	BDL
			R_3	BDL	BDL	BDL	BDL
	T_2	160	R_1	BDL	BDL	BDL	BDL
			R_2	BDL	BDL	BDL	BDL
			R_3	BDL	BDL	BDL	BDL
	T_3	Untreated	R_1	BDL	BDL	BDL	BDL
			R_2	BDL	BDL	BDL	BDL
			R_3	BDL	BDL	BDL	BDL
II	T_1	80	R_1	BDL	BDL	BDL	BDL
(115 DAS)			R_2	BDL	BDL	BDL	BDL
			R_3	BDL	BDL	BDL	BDL
	T_2	160	R_1	BDL	BDL	BDL	BDL
			R_2	BDL	BDL	BDL	BDL
			R_3	BDL	BDL	BDL	BDL
	T_3	Untreated	R_1	BDL	BDL	BDL	BDL
			R_2	BDL	BDL	BDL	BDL
			R_3	BDL	BDL	BDL	BDL

DAS - Days after last spray, BDL - Below detectable limit

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