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## Evaluation of Residues of β-Cyfluthrin on Cotton

I. Mukherjee, M. Gopal, Kusum

Division of Agricultural Chemicals, Indian Agricultural Research Institute, New Delhi 110012, India

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Cotton is the most important fibre crop of India. It is grown in nearly 9 million hectares with an annual yield of about 130.9 lakh bales of 170 kg each with a productivity of 246 bales ha<sup>-1</sup>. India ranks number one in the world in respect of the area of its cultivation and fourth in total production. The crop suffers heavy losses due to insect pest infestation. It is estimated that 22% of the crop yield is lost on account of insect pests infestation amounting to Rs. 20,000 million. Of this, bollworm complex alone causes 50-60% loss and white fly (*Bemisia tabaci*) also affects loss in cotton seed yield. Other major pests are aphids (*Aphis gossypii*), jassids (*Amrasca devastans*) and leaf roller (*Syllepte derogata*).

All India Coordinated Research Project on Cotton has recommended (Agnihotri, 1999) the use of endosulfan and synthetic pyrethroids on cotton crop for the control of bollworm complex, white fly etc. Trials conducted in India and abroad have revealed the effectiveness of synthetic pyrethroids in reducing the incidence of bollworms and increasing the productivity of quality cotton (Ruscoe, 1980; Anonymous, 1999).

β-Cyfluthrin [(SR)- $\alpha$ - cyano - 4 fluoro - 3 - phenoxybenzyl (1RS, 3 RS; 1RS, 3SR)-3- (2,2 -dichlorovinyl) - 2, 2-dimethylcyclopropanecarloxylate] is a relatively new addition to this group (Pap et al., 1996). This insecticide is highly effective against insect pests due to fluorine bonus (Nauman, 1998), yet on a scale of mammalian safety, it is as safe as natural product pyrethrin I. The information for the persistence of β-cyfluthrin on various crops is limited (Sinha, 1998; Battu et al., 1999; Sinha and Gopal, 2001). Considering the extensive use of pesticides on cotton in India, it is essential to evaluate the safety of a new molecule giving emphasis to persistence behaviour of the insecticide. This paper presents the residues of β-cyfluthrin in cotton seed, lint and soil at harvest for three consecutive years of field usage.

## MATERIALS AND METHODS

Supervised field trials were conducted for 3 years consecutively in the field of Indian Agricultural Research Institute, New Delhi using Randomized Block

Design to study the residues of  $\beta$ -cyfluthrin insecticide. The insecticide was applied at the recommended dose of 18.75 g ai ha<sup>-1</sup> and double the recommended dose i.e. 37.5 g ai ha<sup>-1</sup> at the 50% boll formation stage. Analytical standard of  $\beta$ -cyfluthrin (98.7% a.i.) as well as formulation Bulldock 025SC were obtained from M/s. Bayer India and the formulation was tested for its active ingredient by analysis by GLC before use in the field trial. Cotton (var. H 1098) was raised in Delhi following recommended agronomic practices for this region of north India. There were three replications for each treatment and the area of each plot was 25 m<sup>2</sup>. The first application to the crop was made every year at 50% boll formation stage (10 August 1998, 1999 and 2000) followed by three more applications each year at 10 day intervals using knapsack sprayer. A total of four applications of the insecticide was made each year. The amount of spray fluid (water) used was @ 500 L ha<sup>-1</sup>. Harvest time (15. 10. 1998, 15. 10. 1999 and 15. 10. 2000) samples of cotton seed were collected 10 days after the last application for analysis. A control plot of cotton was set aside every year, where no insecticide was applied.

All the solvents like dichloromethane, n-hexane and acetone were distilled in glass apparatus before use. Soil samples from the cotton field collected 10 d after the last application, were air-dried, sieved and then subjected to Soxhlet extraction. The cotton pods were delinted manually to separate cotton seed and lint to analyze them separately. Soil (50 g), finely powdered cotton seed (25 g) and cotton lint (10 g) were separately extracted with n-hexane-acetone (1:1 V/V) for six hours using Soxhlet apparatus.

Cotton lint and soil samples were cleaned before analysis by gas liquid chromatography (GLC). The extract from cotton lint and soil samples was concentrated to 10~mL and passed through a glass column packed with sodium sulfate (2g) + acidic alumina (3 g) + sodium sulfate (2 g). The column was prewashed with hexane (20 mL) and the insecticide eluted with a mixture of hexane + acetone (9:1,V/V). The organic solvent was concentrated to 10~mL and then subjected to GLC analysis.

The cotton seed extract was concentrated and oil obtained was dissolved into n-hexane (30mL) and partitioned into acetonitrile (3 x 30 mL). The combined acetonitrile fraction was treated with 150 mL aqueous sodium chloride solution (150 mL, 3%, W/V) and partitioned into dichloromethane (2 x 100 mL). After concentration and complete evaporation of dichloromethane, the residues were taken in hexane (10 mL) before subjecting to analysis by GLC fitted with electron capture detector.

The operating parameters of GLC were as follows: glass column (2 m length, 2 mm id) filled with 3% SE-30 coated as Chromosorb W.H.P. (80-100 mesh), Temperature for column, injector and detector were 260°C, 270°C and 300°C, respectively. Carrier gas flow (nitrogen) was 30 mL min<sup>-1</sup>. Relative retention time of  $\beta$ -cyfluthrin was 9.25 min. Samples fortified with  $\beta$ -cyfluthrin on cotton soil, cotton seed and cotton lint were processed and analyzed following the method

given above and the average recoveries were 82-87% from spiked samples of seed, 75-79% from lint and 78-80% from soil at 0.5 and 1 mg kg<sup>-1</sup>. No corrections were therefore applied in the results. The minimum detection limit of the pesticide was 0.001 mg kg<sup>-1</sup>.

## RESULTS AND DISCUSSION

The residues of  $\beta$ -cyfluthrin were detected in cotton lint and seeds with average levels varying from 0.001 to 0.003 mg kg<sup>-1</sup> following the application @ 18.75 g ai ha<sup>-1</sup>. Since cotton seed is an oilseed and is also used in animal feed, it is essential to record residues of this new insecticide in this commodity before recommending the insecticide for field usage. Seed samples were found to contain residues of this pesticide in low amounts (ND <0.001 mg kg<sup>-1</sup> to 0.003 mg kg<sup>-1</sup>) when applied at recommended rate for three years consecutively (Table 1).

The maximum residue limit (MRL) of  $\beta$ -cyfluthrin on cotton has been fixed in Mexico as 0.1 mg kg<sup>-1</sup> and by FAO/WHO (1996) for cyfluthrin as 0.05 mg kg<sup>-1</sup>. However, in the present study, the residues of  $\beta$ -cyfluthrin in cotton seed were much below this level (0.05 mg kg<sup>-1</sup>) following recommended dose of the insecticide. The residues were found to be 0.004- 0.0087 mg kg<sup>-1</sup> in seeds on applying double the recommended dose i.e. 1500 mL ha<sup>-1</sup> of formulation (37.5 g a.i. ha<sup>-1</sup>). No MRL is available for  $\beta$ -cyfluthrin on cotton lint as it is not an edible commodity.

The presence of residues in lint could be due to binding of  $\beta$ -cyfluthrin with cellulose present in the lint and/or physical adsorption. Since the lint/cotton fibre and seed are more exposed to abiotic factors, it has dissipated more from these commodities than from soil, wherein the lipophilic insecticide is found to be higher than that recorded in cellulosic lint or cotton seed. The insecticide is not systemic in action hence does not reach the oil seed and get accumulated like that observed with other pesticides like alphamethrin (Tamilselvan, 1995) and monocrotophos in cotton foliage (Cahill et al., 1975) and dimethoate in mustard (Mukherjee and Gopal, 1996). Since most of the pesticide falls on the soil at the time of application, it is natural to record the residues of the insecticide highest in this commodity.

The insecticide  $\beta$ -cyfluthrin was effective in controlling the insect pest population of cotton. The persistence behaviour studies indicated that it did not translocate in the lint and seed at levels higher than the recommended organochlorine insecticide, endosulfan (Agnihotri, 1999).

The fact that only small amounts of  $\beta$ -cyfluthrin residues were recorded in cotton lint and seed at levels lower than the prescribed MRL proves it to be safe from environmental point of view. Hence the schedule can be recommended for plant protection of cotton crop grown in semitropical climate of India.

**Table1.** Residues of β-cyfluthrin in cotton seed, lint and soil under cotton crop.

Substrate	Treatment	1998	1999	2000
Seed	18.75 mg kg <sup>-1</sup>	0.0016	0.0020	0.0030
		$(\pm 0.0006)$	( <u>+</u> 0.0010)	( <u>+</u> 0.00)
	37.50 mg kg <sup>-1</sup>	0.0040	0.0087	0.0064
		(±0.0012)	( <u>+</u> 0.0005)	( <u>+</u> 0.0002)
Soil	18.75 mg kg <sup>-1</sup>	0.0400	0.0866	0.0533
		$(\pm 0.0100)$	(±0.0057)	(±0.0057)
	37.50 mg kg <sup>-1</sup>	0.1530	0.1800	0.1433
		(±0.0152)	(±0.0100)	(±0.0152)
Lint	18.75 mg kg <sup>-1</sup>	0.0010	0.0013	0.0016
		$(\pm 0.0000)$	( <u>+</u> 0.0005)	( <u>+</u> 0.0011)
	37.50 mg kg <sup>-1</sup>	0.0030	0.0023	0.0030
		(±0.0010)	( <u>+</u> 0.0005)	( <u>+</u> 0.0010)

Standard deviation is given in parenthesis

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