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Harvest Time Residues of Emamectin Benzoate in Cotton

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Studies were conducted to evaluate the harvest time residues of emamectin benzoate 5 EC on cotton in Tamil Nadu Agricultural University, Coimbatore during 2005-2007. Two sprays of emamectin benzoate 5 EC at 15 and 30 g a.i. ha^{-1} were given along with untreated check. Samples of cotton lint, seed and soil were collected at random at first and third picking (50 and 80 days after last spray, respectively) for analysis. The results revealed that the harvest time residues of emamectin benzoate 5 EC at 15 and 30 g a.i. ha^{-1} were below detectable level in cotton lint, seed, oil and soil samples.

Key words: Emamectin benzoate, cotton, residue

Cotton (*Gossypium hirsutum* Linn.), an important commercial / fibre crop in India plays a key role in national economy with an export worth of Rs.38, 000 crores (Dhawan, 1998). In India, it is grown under varying climatic and soil conditions in an area of 85.6 lakh ha, with a production of 223 lakh bales. Tamil Nadu accounts for 1.60 lakh ha producing 5.50 lakh bales with a productivity of 584 kg lint ha^{-1} (Raveendran *et al*., 2002). The damage caused by the insect pests is one of the major causes for poor yield of cotton. Nearly 1326 insects and mites all over the world (Hargreaves, 1948) and about 200 in India (Anonymous, 1981) have been recorded as pests of cotton. So the efforts in the past resulted in the development of less persistent chemicals with novel mode of action to overcome the ecological constraints like resurgence, resistance and residues. At present the Golden Age of insecticide research has many selective, neuro active and easily degradable compounds. These newer molecules always have a higher stability and superiority over the conventional pesticides to control the pest population density in classical manner at field level. Emamectin benzoate, one of the newer compounds is synthesized from the naturally occurring insecticide/acaricide of avermectin family. This was discovered in 1984 as a broad spectrum lepidoptericide. Patil and Rajanikantha (2004) reported emamectin benzoate under the new class of insecticide “avermectins” and explained its mode of action and efficacy. This product is a mixture of emamectin benzoate B_1a and emamectin benzoate B_1b that are extracted from *Streptomyces avermitilis*. It is both a stomach and contact insecticide. It interferes with neurotransmitters of target pests which results in disruption of nerve impulses. It is used primarily for control of lepidopteran pests in foliage and fruity vegetables (Ishaaya and Ohsawa, 2002; Leibee *et al*., 1995; Jansson *et al*., 1996), cotton (White *et al*., 1997) and range of other crops (Dunbar *et al*., 1998). Hence the present study was undertaken to evaluate the residue of emamectin benzoate 5 EC in cotton.

Materials and Methods

Emamectin benzoate 5 EC was tested in two doses to evaluate the harvest time residue in cotton during 2005-07 at Tamil Nadu Agricultural University, Coimbatore. The experiments were conducted in a randomized block design with a plot size of 5 x 5m with seven replications. The treatments tried were emamectin benzoate 5 EC@ 15g a.i ha^{-1}, 30 g a.i ha^{-1} and untreated control. Applications of different treatments were imposed three times at an interval of 15 days on

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reproductive stage using a high volume sprayer. The spraying was done during morning hours in such a way as to give uniform coverage on foliage and to avoid drift.

Collection of sample

For pesticide residue analysis, sampling of cotton kapas was done during first and third pickings. The interval between the last spray and the first harvest and the first harvest to third harvest were 50 and 30 days, respectively. The samples were ginned to analyse pesticide residues in seed, lint and soil. The samples of lint, seed and soil were analysed for pesticide residue using HPLC as detailed below.

Analytical methodology

Lint

Ten gram of cotton lint was soaked in 50 ml acetonitrile over night and was blended in a blender for approximately five minutes. The mixture was filtered through filter paper and micro filtration unit using filter paper supported on Buchner funnel in to 500 ml vaccum filter flask. Five ml filtered solution was diluted to 25 ml by acetonitrile mixture (750 ml acetonitrile + 250 ml HPLC water + 0.8 ml triethanolamine (TEA) in a 25 ml volumetric flask. Twenty five microliters of this sample was directly injected to HPLC (Shimadzu LC - 20 AT model) with a running time of 10 minutes. Prior to this a chromatogram was prepared with standard solution of emamectin benzoate with the same configuration.

Seed

Twenty grams of cotton seed was soaked in 50 ml acetonitrile over night and was blended in a blender for approximately five minutes. The mixture was filtered through filter paper and micro filtration unit using filter paper supported on Buchner funnel in to 500 ml vaccum filter flask. Five ml filtered solution was diluted to 25 ml by acetonitrile mixture (750 ml acetonitrile + 250 ml HPLC water + 0.8 ml TEA in a 25 ml volumetric flask. Twenty five microliters of this sample was directly injected to HPLC (Shimadzu LC - 20 AT model) with a running time of 10 minutes.

Oil

Fifty gram of seed was blended, tumbled and placed in Soxhlet apparatus and run for 6-8 h in hexane to extract the oil. Hexane portion was collected, condensed and the oil content weighed. To this 25 ml of acetonitrile mixture (750 ml acetonitrile + 250 ml HPLC water + 0.8 ml TEA) in a 25 ml volumetric flask. Twenty five microliters of this sample was directly injected to HPLC (Shimadzu LC - 20 AT model) with a running time of 10 minutes. Prior to this a chromatogram was prepared with standard solution of emamectin benzoate with the same configuration.

Soil

The soil samples taken from treated plots were air dried. It was ground in a pestle and mortar and passed through 2 mm sieves. Five gram soil sample was taken in a 50 ml volumetric flask and mixed with 25 ml acetonitrile and kept for 48 hrs following sonication for 16 hrs in a sonicator. The solution was filtered and 1 ml aliquot was made up to 50 ml with acetonitrile mixture (750 ml acetonitrile + 250 ml HPLC water + 0.8 ml TEA). Twenty five microlitres of this sample was directly injected to HPLC (Shimadzu LC - 20 AT model) with a running time of 10 minutes.

Final quantification

Emamectin benzoate residues were estimated by Shimadzu LC - 20 AT model HPLC equipped with SPD - M20A prominence Diode array detector (DAD) fitted with RP-18 e Chromolith® column. The following were the operating parameters.

Column : RP- 18e Chromolith®
Temperature : Ambient temp. (40^0C)
Detector : SPD – DAD (Diode array detector
Mobile phase : Acetonitrile + double distilled water + TEA (750 : 250 : 0.8v /v)
Flow rate : 1 ml/min.
Wavelength : 260 nm
Total run time : 10 minutes

The amount of residue was determined by comparing the sample response with the response of standard by using the formula,

$$\text{Residues} = \frac{A_s}{A_{std}} \times \frac{W_{std}}{W_s} \times \frac{V_s}{A_{sj}}$$

Where, A_s - Peak area of the sample, A_{std} - Peak area of the standard, W_{std} - Weight of the standard in ng, Ws - Weight of the sample in g, Vs - Volume of the sample (final extract in ml), Asj - Aliquot of the sample injected in ml

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 emamectin benzoate at 1, 2 and 5 ppm level. The results of the recovery study are presented in Table 1. The mean recovery of emamectin benzoate was 73.2, 74.9, 76.8 and 76.0 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 the sample was 0.04 and 0.1 mg g^{-1}, considering the weight of the sample as 25 and 10 g and final volume of the extract as 2 ml for cotton seed and lint, respectively while that was 0.5 mg g^{-1} for oil considering the sample weight of 2g. The harvest time residues of emamectin benzoate 5 EC at 15 and 30 g a.i.ha^{-1} were at below detectable level in cotton seed, lint and oil samples collected during first and third harvest in both the

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

Matrix / Substrate	Amount fortified (ppm)	% recovered	Average % recovery
Lint	1.0	72.1	
	2.0	73.0	73.2
	5.0	74.5	
Seed	1.0	74.8	
	2.0	73.5	74.9
	5.0	76.4	
Oil	1.0	80.2	
	2.0	77.5	76.8
	5.0	72.6	
Cropped soil	1.0	75.6	
	2.0	74.3	76.0
	5.0	78.0	

***** Average of seven replicates

Table 2. Harvest time residues of emamectin benzoate 5 EC in cotton-Field experiment I and II

Treatment	Residues in µg g^{-1} at harvest							
	Lint		Seed		Soil		Oil	
	I Picking	III Picking	I Picking	III Picking	I Picking	III Picking	I Picking	III Picking
Emamectin benzoate 5 EC @ 15 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Emamectin benzoate 5 EC @ 30 g a.i. ha^{-1}	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Untreated control	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

BDL – Below detectable level

experiments (Table 2). Yoshi *et al*. (2000) reported that the detection limits of the analytes in vegetables were 0.1-0.3 ppt, and nine commercial crops had 0.2- 6.7 ppb of emamectin benzoate out of 20 crops surveyed. Crouch *et al*. (1997) reported that cabbages after treatment with 8 weekly applications of avermectin at 0.015 lb and 0.075 lb a.i. ac^{-1} required pre harvest intervals of 2 h to 10 days; and the total residue declined from 450 to 200 and 2900 to 1300 ppb for the two doses, respectively. Gonzalez and Barria (1999) stated that the residues of abamectin fell below 2.0 ppb after 8 days in many vegetable and fruit crop nectarines. Cobin and Johnson (1995) determined abamectin residues in apples by using reversed-phase liquid chromatography with fluorescence detection. Recoveries of avermectins from apples fortified with about 2-77 ppb avermectin B1a or 2-27 ppb 8, 9-Z avermectin B1a averaged 85 per cent. The limit of quantitation was 2 ppb and the limit of detection was 1ppb for each analyte.

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