Madras Agric. J., 99 (7-9): 580-582, September 2012
https://doi.org/10.29321/MAJ.10.100145

Short Note

Harvest Time Residues of Spirotetramat in Cotton

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Two field studies were conducted to assess the harvest time residues of spirotetramat on cotton at TNAU, Coimbatore during 2006 and 2007. Spirotetramat was sprayed thrice at fifteen days interval at 75 and 150 g a.i. ha^{-1}. Samples of seed, lint and oil were extracted with acetonitrile: water (80:20 v/v, containing 0.22 ml formic acid l^{-1}). The results revealed that the harvest time residues of spirotetramat were below detectable level (BDL) in cotton lint, seed, oil and soil samples in both the field experiments.

Key words: Spirotetramat, Cotton, Residues, HPLC, Kapas

Cotton growers in India depend heavily on synthetic pesticides to combat pests. Most of the insecticides used on cotton belong to organophosphates, carbamates and synthetic pyrethroids. At present, the Golden Age of insecticide research has met with selective, neuro active and easily degradable compounds. These newer molecules have higher stability and superiority over the conventional pesticides to control the pest population density in classical manner at field level.

Spirotetramat (*cis* -4- (ethoxycarbonyloxy) -8- methoxy -3- (2,5-xylyl) -1- azaspiro [4.5] dec-3-en-2-one), is a novel compound derived from tetramic acid and it is the third molecule in ketoenol family (the first and second being spirodiclofen and spiromesifen respectively). It is endowed with a unique mode of action of inhibiting insect lipid biosynthesis and offering broad spectrum control with systemic action against sucking pests of several crops.

This study was carried out to determine the harvest time residues of spirotetramat in cotton samples.

Materials and Methods

Two field experiments were conducted to determine the harvest time residues of spirotetramat on Super Bunny and MCU 5 cotton varieties. The field experiments for residue analysis were conducted from September 2006 to February 2007 and March to June 2007 in the farmer's holding at Sengalipalayam and Nariyampallipudur near Annur, respectively. Spirotetramat was sprayed at 75 and 150 g a.i. ha^{-1} with hand operated knapsack sprayer. Each treatment was replicated five times in a randomized block design. Sampling of cotton kapas was done during first and third pickings. The samples were ginned to analyse residues in the seed, lint and oil.

Extraction

Seed

Twenty five grams of crushed seed sample was extracted with 150 ml acetonitrile: water (80:20 v/v, containing 0.22 ml formic acid l^{-1}) by shaking on a mechanical shaker for half an hour. The contents were filtered through Whatman No.1 filter paper mounted on a Buchner funnel and the filtrate was collected. The extraction flask and contents on filter paper were rinsed with 100 ml acetonitrile. The pooled filtrate was collected into a round bottom flask and concentrated on rotary vacuum evaporator with water bath maintained at 40°C.

Lint

Lint sample (10 g) was extracted with 200 ml acetonitrile: water (80:20 v/v, containing 0.22 ml formic acid l^{-1}) by shaking on a mechanical shaker for half an hour.

Oil and Soil

The oil was extracted from cotton seed by eluting 25 g of finely ground cotton seeds with 250 ml of hexane. Then hexane was removed from oil using rotary vacuum evaporator. An aliquot of 5 g cotton seed oil was dissolved in 30 ml of hexane. The contents were transferred to a 250 ml separating funnel and the residues were extracted with acetonitrile: water (80:20 v/v, containing 0.22 ml formic acid l^{-1}).

The procedure followed for extracting residues from seeds was followed for extraction from soil too except that 100 ml acetonitrile: water was used for rinsing the extraction flask and contents on filter paper.

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Clean up

The concentrated residue obtained from extraction was quantitatively transferred on to a Supelclean™ ENVI™ - Carb SPE tubes. The column was eluted with 30 ml mixture of acetonitrile: water (80:20 v/v, containing 0.22 ml formic acid l_{-1}). The eluate was collected and dried on rotary vacuum evaporator with water bath maintained at 40° C. The residue was dissolved in 5 ml solvent mixture (acetonitrile: water 80:20) and subjected for high performance liquid chromatography (HPLC) analysis.

Final determination

The reference standard of spirotetramat with 99.8 per cent purity and its metabolite spirotetramat enol with 99.4% purity obtained from M/S Bayer Crop Science Ltd, New Delhi were used for quantification and to determine the detectable limits.

Concentrated stock solution

The technical grade spirotetramat with 99.8 per cent purity was adjusted to 100 mg a.i. and transferred to a 100 ml volumetric flask and the volume was made up to 100 ml with acetonitrile. The flask was shaken well to get a homogenous solution of 1000 ppm and was stored in a refrigerator.

Intermediate stock solution

The concentrated stock solution was brought to room temperature and one ml from the concentrated stock solution was transferred to a 100 ml volumetric flask. The volume was made up and shaken well to obtain a homogenous solution of 10 ppm of intermediate stock solution. This 10 ppm solution was utilized for fortification of samples.

Working standard

From the intermediate stock solution, after bringing to room temperature, working standards of 0.5, 1 and 2 ppm were prepared by diluting 0.5, 1 and 2 ml of 10 ppm solution to 10 ml with acetonitrile. These working standards were used to find out the retention time of these compounds and for quantitative determination of residues in samples.

Recovery studies / Fortification

Cotton samples were fortified at 0.2, 0.5 and 1 ppm by adding required quantity of 10 ppm standard solution to work out the recovery per cent of analytical methodology. The samples were homogenized after fortification of standard solution, extracted and subsequently analysed for the residues of spirotetramat and its metabolite enol. Five replicate determinations were made at each fortification level along with two control samples. The limit of quantification was established based on the recovery.

Quantification

Spirotetramat 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
Wavelength	:	260 nm
Mobile phase	:	Acetonitrile : water (80:20)
Flow rate	:	1.0 ml/min.
Total run time	:	10 min

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

$$\text{Residues in ppm} = \frac{H_s}{H_{std}} \times \frac{W_{std}}{W_s} \times \frac{V_{ex}}{V_s} \times \frac{A_s}{A_{std}}$$

where, H_s - Peak height of the sample, H_{std} - Peak height of the standard, W_{std} - Weight of the standard injected in ng, W_s - Weight of the sample in g, V_{ex} - Volume of the final extract in ml, V_s - Quantity of the sample injected in µl, A_s - Attenuation of the sample and A_{std} - Attenuation of the standard.

Results and Discussion

The mean recovery of spirotetramat was 87.60 per cent from fortified lint samples, 87.76 per cent from seed, 86.50 per cent from oil and 84.25 per cent from soil samples at 0.2, 0.5 and 1.0 ppm level. The sensitivity of the instrument was 0.2 ppm and the determinability level in the samples were 0.08, 0.04 and 0.016 µg g_{-1} considering the weight of the cotton seed oil (5g), lint (10 g) and cotton seed (25 g), respectively and final volume of the extract as 2 ml. The harvest time residues of spirotetramat 150 OD at 75 and 150 g a.i. ha_{-1} as foliar spray were below detectable level (BDL) in cotton lint, seed and oil samples of first and third pickings in both the field experiments. The interval between the last spray and first picking was 46 and 78 days in the field experiments I and II, respectively. This is in concordance with results of Pandiselvi *et al.,* (2010), who reported that residues of imidacloprid and spirotetramat on cotton plant dissipated to below the detectable levels by tenth day. Analysis of the samples collected at hatvest showed no detectable residues of imidacloprid, spirotetramat and its metabolite enol in cotton lint, seed oil and soil. The pre harvest interval established by these authors was 25 days. Kumar (1998), Suganthy (2003) and Preetha (2007) also found that the residues of imidacloprid in cotton lint, seed, oil and soil were below detectable levels. The present results are similar to the findings of Kavitha (2003), Suganyakanna (2006) and Thilagam (2006) who reported that residues of spiromesifen, acetamiprid and flubendiamide, respectively were not detected in harvest time samples of lint, seed and oil in cotton.

References

Kavitha, J. 2003. Evaluation of spiromesifen 240 SC (Oberon®) against yellow mite of chilli and whitefly of cotton and determination of residues. M.Sc. (Ag.) Thesis, Tamil Nadu Agric. Univ., Coimbatore, India, 96p.

Kumar, K. 1998. Studies on bioefficacy and determination of residues of imidacloprid applied against sucking pests of cotton. Ph.D. Thesis, Tamil Nadu Agric. Univ., Coimbatore, India, 114p.

Pandiselvi, S., Sathiyanarayanan, S. and Ramesh, A. 2010. Determination of spirotetramat and Imidacloprid residues in cotton seed, lint, oil and soil by HPLC UV method and their dissipation in cotton plant. *Pestic. Res. J*. **22**: 168-173

Preetha, G. 2007. Impact assessment of chloronicotinyls on the management of sucking pests in cotton and bhendi ecosystem. Ph.D. Thesis. Tamil Nadu Agric. Univ., Coimbatore, India.

Raveendran, T.S., Mohan, S., Ravikesavan, R., Surendran, C., Meyyazhagan, K. and Parthiban, V.K. 2002. Cotton-The White gold. Tamil Nadu Agricultural Univ., Coimbatore, India. pp. 1-8.

Suganthy, M. 2003. Bioefficacy and residues of Confidence ® (Imidacloprid 17.8% SL) on cotton, vegetables and mango. Ph.D. Thesis. Tamil Nadu Agric. Univ., Coimbatore, India, 197p.

Suganyakanna, S. 2006. Evaluation of acetamiprid 20 SP against sucking pest complex in cotton. Ph.D. Thesis. Tamil Nadu Agric. Univ., Coimbatore, India, 190p.

Thilagam, P. 2006. Evaluation of flubendiamide 480 SC against bollworm complex in cotton and leaf folder and stem borer in rice. Ph.D. Thesis. Tamil Nadu Agric. Univ., Coimbatore, India. 232p

Received: January 18, 2012; Accepted: May 9, 2012