



Short Note

Harvest Time Residues of Abamectin 1.9 EC in Cotton Seed, Lint and Oil

R. Sheeba Jasmine¹, S. Kuttalam² and J. Stanley³

¹Sugarcane Research Station, Melalathur-635 806

²Department of Agricultural Entomology, TNAU, Coimbatore

³Vivekananda Institute of Hill Agriculture, ICAR, Almora - 263 601, Uttarakhand

Harvest time residues of abamectin 1.9 EC after four applications at 14 days interval on cotton @ 10.8, 14.5, 18.5, 22.5 and 29.0 g a.i.ha⁻¹ were determined in seed, lint and oil using high performance liquid chromatography (HPLC) equipped with F-1050 fluorescence detector. The interval between the last spray and first harvest was 37 and 34 days in the first and second field experiments, respectively. The harvest time residues of abamectin were at below detectable level (BDL) in cotton seed, lint and oil sample at all the concentrations tested.

Key words: Abamectin 1.9 EC, harvest time residues, cotton seed, lint, oil, high performance liquid chromatography, fluorescence detector

Cotton crop is attacked by several insect pests at all the stages of crop growth. Among them, bollworms such as *Helicoverpa armigera* (Hubner), *Earias vittella* F. and *Pectinophora gossypiella* (Saunders) are major threats to cultivation and drastically reduce its yield. Abamectin is a broad spectrum pesticide belonging to avermectins group with good contact, stomach as well as partial systemic action. It has been reported to possess excellent performance against the pests of cotton and vegetables (Lima *et al.*, 1994, Bellettini *et al.*, 1999 and Weintraub, 2001). Chemical insecticides and their indiscriminate use have caused a number of problems such as pests developing resistance to insecticides, pest resurgence, and bio concentrations of pesticide residues in consumable produce at harvest. Among the harmful effects of insecticides, persistence of toxic pesticide residues in plants, soil and water are of great concern for consumer's health and safety to animals. Further, they also adversely affect soil health, aquatic life and quality of drinking water. Whenever a chemical is recommended, the harvest time residues need to be determined for long term usage of the compound. Hence, the present study was planned to determine the terminal residues of abamectin 1.9 EC in cotton seed, lint and oil following its repeated application (4 times) on cotton crop.

Materials and Methods

The residues were determined in cotton samples as per the method suggested by Diserens and Henzelin (1999) with slight modification.

Chemicals

Trifluoro acetic anhydride, 1- methylimidazole

and triethylamine and HPLC grade solvents like acetonitrile, petroleum ether and double distilled water were used for the study.

Reference standard

The reference standard of abamectin with 95.2 per cent purity obtained from M/s. Jaishree Agro Industries Ltd, New Delhi was used for quantification.

Field experiments

Two field experiments were conducted one each at farmers holding, Rudhirampalayam and Eastern block, Tamil Nadu Agricultural University, Coimbatore with Rajath and MCU 12 cotton varieties, respectively according to the recommended agronomic practices. The experiments were carried out in plots of 4 x 5 m (20m²) size in a randomized block design with six treatments, each replicated four times. The treatments were imposed four times at 14 days interval commencing from 60th day after sowing. Applications were made during morning hours to avoid photo oxidation of the insecticides. Cotton plants were sprayed with abamectin 1.9 EC at the rate of 10.8, 14.5, 18.5 22.5 and 29.0 g a.i ha⁻¹ with the help of pneumatic knapsack sprayer using 1000 litres of spray fluid per hectare.

Sampling

Cotton lint and seed samples were collected from each replicate of five treatments at first, second and third harvests. Samples of 500g were collected from all the four replications for each treatment, pooled together, and after quartering, a sub samples of 10g of cotton lint and 25 g of seed in duplicate were drawn for lab analysis. Control samples were collected similarly from untreated plots.

¹Corresponding author email: shepris2000@yahoo.com

Extraction

a. Lint

Ten gram of sample was soaked in 300 ml of HPLC grade acetonitrile in a 500 ml storage bottle for 24 h. Then the lint sample was filtered through filter paper supported on a Buchner funnel into a 500 ml vacuum filter flask and washed with 100 ml acetonitrile. After pressing the residual solvent from the solids using rubber damming, the solids were discarded and the final extract was condensed to 40 ml. Finally, 50 μ l of triethylamine was added and the volume made upto 50 ml with acetonitrile. Then clean up step was followed.

b. Seed

The sample of cotton seed (25 g) was soaked in 100 ml of HPLC grade acetonitrile in a 500 ml storage bottle for 24 h, homogenized and filtered through Buchner funnel. After repeated washings, the pooled acetonitrile extract was condensed to 40 ml and 50 μ l of triethylamine was added. Finally, the volume was made upto 50 ml with acetonitrile before clean up.

c. Oil

Twenty five gram of seed was blended, tumbled and placed in a Soxhlet apparatus and extracted for six hours in HPLC grade petroleum ether. Petroleum ether portion was collected and double distilled to recover the oil. Two gram of oil was weighed, 30 ml of acetonitrile added and kept for two hours. Then the contents were transferred to a 50 ml graduated cylinder. Sufficient AR grade acetonitrile was added to make up the volume to 40 ml. After the addition of 50 μ l of triethylamine, the volume was made upto 50 ml with acetonitrile and clean up was followed.

Clean up

The C₁₈ Bond - Elut cartridge was pre washed with 5 ml of HPLC grade acetonitrile and then with 5 ml of conditioning solution. The conditioning solution was prepared by adding 30 ml of acetonitrile, 70 ml of double distilled water and 0.1 ml triethylamine and mixed well. The final test portion extracts (50 ml) were transferred to the C₁₈ Bond - Elut cartridge fitted with adapter and 50 ml reservoir. The sorbent was not allowed to dry during conditioning and before applying the sample. The first eluate was discarded. The cartridge was allowed to dry partially for 10 min, eluted again with 5 ml of acetonitrile and collected in 5 ml amber vials. The resultant extract was condensed to near dryness by using a flash vacuum evaporator at 50°C and taken in reaction vial for derivatization.

Derivatization

Derivatization reagent 1

One volume of trifluoro acetic anhydride was added with two volumes of HPLC grade acetonitrile

in a brown-glass flask. The solution was kept at +4°C.

Derivatization reagent 2

One volume of 1-methylimidazole was added with one volume of HPLC grade acetonitrile in a brown-glass flask. The solution was kept at +4°C.

300 μ l of the derivatization reagent 1 and 200 μ l of derivatization reagent 2 were added in the reaction vial and mixed well. The final determination was made using in high-performance liquid chromatography (HPLC) with fluorescence detector.

Operation parameters of HPLC

Abamectin residues were estimated by Hitachi L-6200 model high performance liquid chromatography (HPLC) equipped with F-1050 fluorescence detector fitted with RP-18 Lichrocart column (25 cm x 4 mm, 5 μ m). The operating conditions were as follows: Temperature: Ambient temp. (35°C), Detector: Fluorescence detector (Xenon source, without pulsating) Excitation: 365 nm, Emission: 470 nm, Mobile phase: Acetonitrile - water (94: 6, v/v), Flow rate: 1.5 ml min⁻¹, Retention time: 8.24 min, Total run time: 20 min.

The final quantification was worked out 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; W_s - Weight of the sample in g; V_s - Volume of the sample (final extract in ml); and A_{sj} - Aliquot of the sample injected in μ l.

Recovery study

Recovery studies using control samples of cotton seed, oil and lint were conducted by fortifying with known quantities of abamectin @ 0.5, 1.0 and 2.0 μ g g⁻¹ to find out the efficiency of the analytical methodology. The minimum detection limit of the instrument was 0.5 ppm and the determinability level in the sample was 0.04 and 0.1 μ g 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 μ g g⁻¹ for oil considering the sample weight of 2g. The mean recovery was 80.80 per cent from samples fortified at 0.5, 1 and 2 μ g g⁻¹ level.

Results and Discussion

The results revealed that the harvest time residues of abamectin 1.9 EC at 10.8, 14.5, 18.5, 22.5 and 29.0 g a.i.ha⁻¹ were at below detectable level (BDL) in cotton seed, lint and oil samples collected during the first, second and third harvest in the first and second field experiments. The interval

between spraying and the first harvest was 37 and 34 days in the first and second field experiments, respectively.

The observed BDL at both the field experiments might be due to faster degradation and sufficient time interval between application and harvest. These results indicating rapid dissipation of abamectin were comparable with the study of Banhawy and Bagoury (1985) who reported the short residual effect of abamectin. Lasota and Dybas (1991) stated that abamectin residues in/on crops were very low, recording less than 0.025 ppm and also observed the half life of abamectin at sunlight as 12 h. Kain and Agnello (2001) reported that abamectin residues dissipated quickly, while Dinesh (2004) concluded that the residues of abamectin at 0.00045, 0.0009 and 0.0012 per cent reached BDL on 3 days after treatment on okra fruits.

References

- Banhawy, E.E.M. and El-Bagoury, E. 1985. Toxicity of avermectin and fenvalerate to the eriophyid mite *Eriophyes dioscoridis* and the predaceous mite *Phytoseiulus finitimus* (Acari: Eriophyidae: Phytoseiidae). *Int. J. Acarol.*, **11**: 237-240.
- Bellettini, S., Ulbrich, A.V. and Gastaldi, L.F. 1999. Control of white mite *Polyphagotarsonemus latus* Banks, with different doses of chlorfenapyr on cotton. In: Anais- II Congresso Brasileiro de Algodao: O algodao no seculo XX, perspectivara para o seculo XXI, Ribeirao Preto, SP, Brasil, 5-10 Setembro 1999, 152-154p.
- Dinesh, K. 2004. Evaluation of abamectin 1.8 EC and profenofos 50 EC against two spotted spidermite and fruit borer on okra. M.Sc. (Ag) Thesis, Tamil Nadu Agric. Univ., Coimbatore-3, India. 129p.
- Diserens, H. and Henzelin, M. 1999. Determination of abamectin residues in fruits and vegetables by high-performance liquid chromatography. *J. Chromatogr.* **833**: 13-18.
- Kain, D. and Agnello, A. 2001. Scaffolds update on pest management and crop development. *Fruit J.*, **10**: 32-36.
- Lasota, J.A. and Dybas, R.A. 1990. Abamectin as a pesticide for Agricultural use. In: O-Noco Proc. Symposium on Onchocerciasis, Leiden, Netherlands. *Acta Leidensia*, **59**: 217-225.
- Lasota, J.A. and Dybas, R.A. 1991. Avermectins: A novel class of compounds: Implications for use in arthropod pest control. *Ann. Rev. Entomol.*, **36**: 91-117.
- Lima, J.O., De, G. and Machado, W.A. 1994. Effectiveness of abamectin against leafminer (*Liriomyza* sp.) (Diptera: Agromyzidae) in tomatoes. *Revista Ceres*, **41**: 559-566.
- Weintraub, P.G. 2001. Effects of cyromazine and abamectin on the pea leaf miner *Liriomyza huidobrensis* (Diptera: Agromyzidae) and its parasitoid *Diglyphus isaea*. (Hymenoptera: Eulophidae) in potatoes. *Crop Prot.*, **20**: 207 - 213.