Residues of thiamethoxam 25 WG (ACTARA®) in plant matrices and soil after application to the cotton ecosystem

V.G. Mathirajan and A. Regupathy
Department of Entomology, Tamil Nadu Agricultural University, Coimbatore INDIA
Correspondence author regupathyra@yahoo.com; mathirajan@mailcity.com
ABSTRACT

The residues of thiamethoxam 25 WG in plant matrices of cotton and in soil was determined by following a solid phase clean up method. The residues in plant matrices viz., cotton lint and seed were below a detectable level. The residues in soil collected from cotton fields were detected up to 4 days after treatment and the level ranged from 0.064 to 0.071, 0.067 to 0.072 and 0.065 to 0.094 ppm at 25, 50 and 100 g a.i./ha respectively.

Introduction

Cotton is the most important natural fiber crop in the world grown over 80 countries in about 32.5 million hectares. Among the cotton growing countries, India has the largest area of nine million hectares and it is grown under diverse agroclimatic conditions (AICCIIP, 2000). Insect pests are the major limiting factors in reducing the production to the extent of 60-70 percent. Pesticide use estimate in India indicates that 55 percent of the insecticides are used in cotton alone though the cotton area is only five percent of total cropped area.

Indiscriminate and excessive use of broad-spectrum insecticides has resulted in insecticide resistance, destruction of natural enemies, resurgence of minor pests, environmental pollution and other socio-economic problems. From the environmental safety point of view, a pesticide applied should disappear after the desired period of pesticidal activity, but in practice, it seldom happens. Parts of the pesticides sometimes remain on cotton until harvest and may persist in produce even after processing.

Besides in produce, pesticides, which fall on the ground during spray application, contaminate the soil. The contamination with persistent pesticides further leads to water pollution by surface run off, sedimentary transport and prolonged drainage. The extent of contamination depends on the nature of the pesticide and matrix in which it is present (Agnihotri and Gajbhiye, 1999). Thus, the extensive and excessive contamination of cotton produces and soil with pesticide residue pose a serious threat to man and his environment. There are several reports on presence of various insecticides residues in economic produces of cotton viz., lint, seed and its bi-products cake and oil. Newer and safer insecticides or formulations are being developed in recent years with a view to achieve better and less hazardous pest control. Thiamethoxam (3-(chlorothiazol-S-methyl)-5 methyl (1,3,5) oxadiazinan-4- ylde-N-nitroamine) is a new generation chloronicotinyl compound, which is known to possess desirable insecticidal properties against many homopteran species (Mathirajan and Regupathy, 2002).

However there are very few reports on the residues of this insecticide on crops on which it has been recommended. Thus, there is a need to develop a sensitive and reliable method for estimation of this compound in various economic products and in environmental samples.

The present study was taken conducted estimate the residues of thiamethoxam 25 WG in/on plant matrices viz., lint, seed and in soil.

Materials and Methods

Two field trials (winter 2000 and summer 2001) were conducted at Kallapuram and gobichettypalayam of Coimbatore and Erode districts of Tamil Nadu for estimation of residues of thiamethoxam in plant matrices and in soil. The experiment was conducted in a randomized block design with six replications. Cotton crop (variety LRA 5166) was sprayed with three rounds of thiamethoxam 25 WG at 25, 50 and 100 g a.i./ha at 30 days interval starting from 30 days after sowing by using a pneumatic knapsack sprayer applying 500 liters/ha for the control of early season sucking pests.

Sampling

Cotton lint (50 g) and seed samples (50 g) were collected from first (120 days after sowing) and third pickings (130 days after sowing) from each plot. Soil samples (50 g) were also collected 1, 4, 10, 14, 30 days after the third spraying (90 days after sowing) from the respective treatments.

Extraction

**Lint** Weighed samples of first and third pickings (10 g) were soaked overnight in 50 ml of a mixture of water plus methanol (1 vol. + 1 vol.). Then the sample was filtered through a Buchner funnel. The pooled extract was condensed in a rotovacuum evaporator and used for final determination without further clean up.

**Seed** Twenty-gram seed samples from the first and third pickings were blended in a Waring blender and residues were extracted with methanol in soxhlet apparatus for six hrs. The pooled extract was condensed in a rotovacuum evaporator.

**Soil** The sub samples of soil (20 g) were equilibrated with 70 ml of a mixture of water and methanol (1 vol. + 1 vol.) overnight. The homogenized soil samples were filtered through a Buchner funnel. The filter cake and extraction jar were washed with the extraction solvent.

Cleanup by solid phase extraction

An aliquot of 5 ml of the filtered extract was diluted with twice that volume of water (10 ml). For soil sample an aliquot of 10 ml filtered extract was concentrated to about 7 ml and this concentrate was diluted with water to a final volume of 10 ml. Phenyl solid phase extraction cartridge (obtained from Varian India Ltd., Mumbai) was conditioned with 3 ml methanol and
3 ml water. The diluted filtrate was percolated through the cartridge and the elute was discarded. The cartridge was washed with 2 ml of water and the elute was discarded. The ENV! carb (obtained from Varian India Ltd., Mumbai) cartridge was conditioned with 3 ml methanol and 3 ml water. The phenyl cartridge was attached to the top of the ENV! carb cartridge with an adapter. The analytes from the upper onto the lower cartridge were eluted with 3 ml, water + methanol (1 vol. + 1 vol). Phenyl cartridge was removed and discarded. The analytes were eluted from the ENVI carb cartridge with 30 ml methanol plus acetonitrile (1 vol. + 3 vol) and the eluates were collected. The combined eluates were evaporated down to about 0.5 ml on the water bath. The residues were diluted to a volume of 5.0 ml with methanol (HPLC grade) and fed into HPLC.

**Final determination of residues**

Thiamethoxam residues in samples were estimated by High Performance Liquid Chromatography (HPLC) Hitachi model L6200 with the following operating parameters.

- **Mobil phase:** Water : methanol (8:2 v/v)
- **Column:** ODS.2
- **Flow rate:** 1 ml/ min
- **Wave length:** 270 nm – thiamethoxam
  256 nm – metabolite

**Results and discussion**

**Determinability**

The minimum detection limit of the instrument was 0.5 mg. The determinability level was 0.125 mg in cotton lint, seed and soil considering 10 g lint sample and 2.5 ml of extract and 20 g seed and soil sample and 5 ml of sample extract.

**Recovery**

The mean recovery was 85.5 percent from cotton lint, 86.5 percent from seed, 84.5 percent from soil.

**Residues in plant matrices**

The residues of thiamethoxam in the samples of cotton collected 30 days after last treatment (90 days after sowing) were below detectable level.

**Residues in soil**

Residues of thiamethoxam applied to cotton in soil varied from 0.065 to 0.094 ppm 1DAT, 0.062 to 0.083 ppm 4DAT and BDL to 0.064 ppm 10 DAT in winter 2000 and respective levels in the summer 2001 were 0.071-0.092 ppm, 0.064 - 0.086 ppm and BDL-0.065 ppm. Residues of thiamethoxam in soil samples collected 14 and 30 days after treatment were below detectable limit.

The residues of thiamethoxam in plant matrices

3 ml water. The diluted filtrate was percolated through the cartridge and the elute was discarded. The cartridge was washed with 2 ml of water and the elute was discarded. The ENV! carb (obtained from Varian India Ltd., Mumbai) cartridge was conditioned with 3 ml methanol and 3 ml water. The phenyl cartridge was attached to the top of the ENV! carb cartridge with an adapter. The analytes from the upper onto the lower cartridge were eluted with 3 ml, water + methanol (1 vol. + 1 vol). Phenyl cartridge was removed and discarded. The analytes were eluted from the ENVI carb cartridge with 30 ml methanol plus acetonitrile (1 vol. + 3 vol) and the eluates were collected. The combined eluates were evaporated down to about 0.5 ml on the water bath. The residues were diluted to a volume of 5.0 ml with methanol (HPLC grade) and fed into HPLC.

**Final determination of residues**

Thiamethoxam residues in samples were estimated by High Performance Liquid Chromatography (HPLC) Hitachi model L6200 with the following operating parameters.

- **Mobil phase:** Water : methanol (8:2 v/v)
- **Column:** ODS.2
- **Flow rate:** 1 ml/ min
- **Wave length:** 270 nm – thiamethoxam
  256 nm – metabolite

**Results and discussion**

**Determinability**

The minimum detection limit of the instrument was 0.5 mg. The determinability level was 0.125 mg in cotton lint, seed and soil considering 10 g lint sample and 2.5 ml of extract and 20 g seed and soil sample and 5 ml of sample extract.

**Recovery**

The mean recovery was 85.5 percent from cotton lint, 86.5 percent from seed, 84.5 percent from soil.

**Residues in plant matrices**

The residues of thiamethoxam in the samples of cotton collected 30 days after last treatment (90 Days after sowing) were below detectable level.

**Residues in soil**

Residues of thiamethoxam applied to cotton in soil varied from 0.065 to 0.094 ppm 1DAT, 0.062 to 0.083 ppm 4DAT and BDL to 0.064 ppm 10 DAT in winter 2000 and respective levels in the summer 2001 were 0.071-0.092 ppm, 0.064 - 0.086 ppm and BDL-0.065 ppm. Residues of thiamethoxam in soil samples collected 14 and 30 days after treatment were below detectable limit.

**References**