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Dissipation patterns of insecticides in okra (*Abelmoschus esculentus*)

Deepak. S[1*] and Shashi Bhushan V[2]

[1*]Directorate of Plant Protection Quarantine & Storage, Faridabad, India

[2]Former Professor, Department of Entomology, Agriculture of College, Hyderabad, India

***Corresponding author: deepaksihimoge@gmail.com**

Abstract

A field experiment was conducted at Students' Farm, College of Agriculture, Hyderabad to evaluate the efficacy of six different insecticides *viz.,* bifenthrin 10 EC at 80 g a.i/ha, fipronil 5 SC at 500 g a.i/ha, flubendiamide 480 SC at 60 g a.i/ha, quinalphos 25 EC at 350 g a.i/ha, profenofos 50 EC at 400 g a.i/ha and beta-cyfluthrin 25 SC at 18.75 g a.i/ha against insect pests of okra. Among them, Profenophos, Beta-cyfluthrin and Flubendiamide were found effective against leafhopper, whitefly and shoot and fruit borer, respectively. Hence, dissipation studies of Profenophos, Beta-cyfluthrin and Flubendiamide were carried out at Laboratory of AINP on Pesticide Residues, Hyderabad by collecting okra fruits at 0, 1, 3, 5, 7, 10 and 15 days. The initial deposits of flubendiamide, prophenophos and beta-cyfluthin were recorded to be 1.49, 1.52 and 0.11 mg kg^{-1}, respectively and dissipated to below detectable level (BDL) on the 5th, and 10th day, respectively. The half-life worked out for flubendiamide, profenofos and beta-cyfluthrin were 1.83, 2.16 and 1.13 days with waiting periods for a safe harvest of the okra fruits were 4.19, 5.10 and 2.62 days, respectively.

Introduction

There are more than 1000 pesticides used around the world to ensure food is not damaged or destroyed by pests. Each pesticide has different properties and toxicological effects.As pesticides are intrinsically toxic and deliberately spread in the environment, the production, distribution and use of pesticides require strict regulation and control.

World Health Organization has two important objectives in respect of pesticides. The first one is to ban pesticides that are most toxic to humans and remain for the longest time in the environment. And second is to protect public health by setting maximum limits for pesticide residues in food and water.

The acceptable daily intakes used by governments and international risk managers, such as the Codex Alimentarius Commission to establish Maximum Residue Limits (MRLs) for pesticides in food. Codex standards are the reference for the international food trade, so that consumers everywhere can be confident that the food they buy meets the agreed standards for safety and quality, no matter where it is produced. Currently, there are Codex standards for more than 100 different pesticides (WHO, 2022). In this background, this experiment was conducted to determine the persistence pattern of a few commonly used and new insecticide molecules in okra (*Abelmoschus esculentus).*

Materials and Method

An experiment was conducted at Students' Farm of Agriculture College, Hyderabad, India, to evaluate the efficacy of six different insecticides *viz.,* bifenthrin 10 EC at 80 g a.i/ha, fipronil 5 SC at 500 g a.i/ha, flubendiamide 480 SC at 60 g a.i/ha, quinalphos 25 EC at 350 g a.i/ha, Profenophos50 EC at 400 g a.i/ha and beta-cyfluthrin 25 SC at 18.75 g a.i/ha against on insect pests of okra.

Among them, Profenophos at 400 g a.i/ha, Beta-cyfluthrin at 18.75 g a.i/ha and Flubendiamide at 60 g a. i/ha was found effective against leafhopper (*Amrasca biguttula*), whitefly (*Bemisia tabaci*) and shoot and fruit borer (*Earias vittella*) respectively.

Further, dissipation studies of Profenophos, Beta-cyfluthrin and Flubendiamide was carried out at the Laboratory of All India Network Project on Pesticide Residues, Hyderabad. Fruits samples size of 250g was collected from the insecticide-treated plots in polythene bags at an interval of 0, 1, 3, 5, 7, 10 and 15 days after the last spray and brought to the laboratory for residue analysis.

Analysis of Residues

Preparation of standard stock solution

From the technical grade of insecticide, one ppm standard solution was prepared by diluting with n-hexane for profenophos and with acetonitrile for flubendiamide and beta-cyfluthrin and used for carrying out recovery and comparative studies of pesticide residues in the fruit samples collected at different intervals.

Extraction and clean up

Prophenophos: A representative sample (okra fruits) of 25 g was homogenized with 50 ml acetonitrile and was filtered. Then the filtrate was evaporated to near dryness using a vacuum rotary evaporator and the contents were re-dissolved in 25 ml of hexane. Then recovered filtrate was partitioned after adding 100 ml of acetonitrile and 125 ml of 5 percent sodium chloride solution. The extract was cleaned up with florosil column eluting with dichloromethane. The elute was concentrated again and dissolved in n-hexane and later subjected to alumina column clean up. The final elution was done with hexane: acetone (9:1) and evaporated to dryness and analyzed on GC. Residues were estimated by comparing the peak area of the standard with that of the peak in the sample under identical conditions.

Flubendiamide**:** A representative sample (okra fruits) of weight 25 g was extracted with 50 ml acetonitrile containing 0.01per cent hydrochloric acid twice by using a mechanical shaker for 30 minutes. The extraction mixture was filtered and evaporated to near dryness using a vacuum rotary evaporator and the contents were re-dissolved in 20 ml of acetonitrile.

A glass column was packed with 5 g of alumina using hexane as a solvent and drained the excess of solvent. The sample was transferred into the column and eluted with 50 ml of 10:1 hexane: ethyl acetate solvent mixture.

Discarded the first 10 ml fraction and collected the elute over anhydrous sodium sulphate, the process was repeated thrice. Approximately 150 ml of elute was collected. The elute was completely dried or drained and evaporated to near dryness. The residues were recovered in a 5ml volume of acetonitrile for HPLC analysis. Residues were estimated by comparison of peak area of the standards with that of the unknown or spiked samples run under identical conditions.

Beta-cyfluthrin: A representative fruit sample of 25 g was homogenized with 50 ml acetone: hexane (1:9) and was filtered. The filtrate was partitioned after adding saturated Nacl and Dichloromethane. The extract was cleaned up with florosil column eluting with hexane. The elute evaporated to dryness for Gas Chromatography analysis. Residues were estimated by comparing the peak area of the standard with that of the peak in the sample under identical conditions.

Fortification and Recovery Studies

Before sample analysis, recovery tests were conducted. For the recovery test, 25 g of okra fruit samples were collected from control plots and fortified at two levels separately i.e. 0.01 ppm and 0.10 ppm for profenofos, flubendiamide and beta-cyfluthrin from the standards prepared. The contents were mixed thoroughly and the samples were extracted and cleaned up as per the procedure described above. All the recovery samples were replicated thrice.

The mean percent recovery of Profenophos in the okra fruit sample was 88 and 87, flubendiamide was 87 and 88 and beta-cyfluthrin was 86 and 88, respectively at 0.01 ppm and at 0.10 ppm level of fortification for each insecticide.

Estimation

The residues of profenofos and beta-cyfluthrin were estimated using Gas Chromatograph - Electron Capture Detector (GC-ECD). Similarly, residues of flubendiamide were estimated using High-Performance Liquid Chromatography (HPLC). Residues were estimated by comparing the peak area of the standard with that of the peak in the sample under identical conditions.

Interpretation of Data

Residues (mg/kg)

The formula used to arrive at the residues is as follows.

$$\frac{\text{Area of sample}}{\text{Area of standard}} \times \frac{\mu\text{l of sample injected}}{\mu\text{g of standard injected}} \times \frac{\text{Final volume}}{\text{Weight of the Sampling}} \times \frac{100}{\text{Per cent mean recovery}}$$

Dissipation (%)

$$\text{Per cent dissipation} = \frac{\text{Initial deposit} - \text{Residues at given time}}{\text{Initial deposit}} \times 100$$

Prediction of approximate time required to dissipate the residue below the tolerance limit

The period to be allowed to expect the residues to reach below the tolerance limit after treatment for safe use of the treated material was calculated by using the formula (Gunther and Blinn, 1955).

$$Y = a + bX$$

Where,

Y = Log of tolerance limit
a = Log of initial deposit
b = Slope of the regression line

Waiting Period: the minimum number of days to lapse before the insecticide reaches the tolerance limit. The waiting periods were calculated by the following formula.

$$T_{tol} = \frac{[a - \text{Log tol}]}{B}$$

Where,

T_{tol} = Minimum time (in days) required for the pesticide residue to reach below the tolerance limit.
a = Apparent initial deposits obtained in the regression equation
tol = Tolerance limit of the insecticide
b = Slope of the regression line

Calculation of Half-life Values (RL_{50}): time in days required to reduce the pesticide residues to half of its initial deposits.

$$RL_{50} \text{ (or) } t_{1/2} = \text{Log } 2 / K_1 = 0.301/K_1$$

K_1 = Slope of regression line (Hoskins, 1961)

Results and Discussion:

Flubendiamide

The initial deposit and subsequent residues of flubendiamide in okra fruit sample was 1.49 mg/kg at 0 days, which gradually dissipated to 0.84, 0.47, 0.10 and 0.01 mg/kg at 1, 3, 5 and 7 days, respectively. The residues fell below maximum residue limit (MRL) of 0.2 mg kg^{-1} in 4days after the treatment. The half-life (RL_{50}) of flubendiamide was worked to be 1.83 days.

The results are in agreement with the findings of Sahoo *et al.* (2009) who evaluated that the average initial deposits of flubendiamide on chilli to be 1.06 and 2.00 mg/kg, respectively, following two applications of flubendiamide 480 SC at 60 and 120 g a.i/ha at 10 days interval. Residues of flubendiamide dissipated below detectable level of 0.01 mg/kg in 7 and 10 days at single and double dosages, respectively (Table 1).

Prophenophos

Initial deposits of Profenophos in okra fruit sample was 1.52 mg/kg at 0 days and further degraded to 1.14, 0.75, 0.62 and 0.09 mg/kg after 1, 3, 5 and 7 days, respectively. The results showed that the residues of Profenophos reached below tolerance limit of 0.5 mg/kg in seven days while corresponding half-life was worked out to be 2.16 days.

The results are in agreement with the findings of Sahoo *et al.* (2004) who reported an initial deposit of 1.37 mg/kg following application of Profenophos at 500 g a.i/ha on tomato. These levels were reduced to below detectable level (BDL) after 15 days of application.

Beta-cyfluthrin

The initial deposit of beta-cyfluthrin (18.75 g a.i. ha^{-1}) in okra was 0.11 mg/kg and at one day after treatment itself around 45.04 per cent dissipation was recorded. Further very rapid dissipation was evident by third day was noticed and initial deposit dissipated to 80.95 per cent by third day. The residues at the end of first and third day were 0.06 and 0.02 mg/kg respectively. The residues of beta-cyfluthrin dissipated to below tolerance limit of 0.02 mg/kg in three days.

The results are in conformity with those of Singh and Singh (2007) who studied the dissipation of beta-cyfluthrin on chickpea following foliar applications at 12.5 and 25 g a.i/ha, recording initial deposit of beta-cyfluthrin 0.109 and 0.135 mg/kg in green pods which dissipated to 88.1-92.6% with half-life of 3.34 and 4.01 day, respectively.

The variation in the rate of dissipation in vegetables may be due to changes in the crop matrix. In addition, the dissipation of pesticide residues in/on crops depends on climatic conditions, type of application, plant species, dosage, interval between application and time of harvest.

Summary

Insecticides *viz.*, flubendiamide (60 g a.i/ha), Profenophos (400 g a.i/ha) and beta-cyfluthrin (18.75 g a.i/ha) sprayed on okra were dissipated more than 90 per cent of its original concentration after seventh day of spray and are safe for harvesting and consumption after that.

Table 1. Dissipation of Flubendiamide, Profenophos andBeta-cyfluthrinin okra

Day	Residues / Deposits (mg/kg)			Dissipation (in percentage)		
	Flubendiamide	**Profenophos**	**Beta-cyfluthrin**	**Flubendiamide**	**Profenophos**	**Beta-cyfluthrin**
0	1.49	1.52	0.11	0	0	0
1	0.84	1.14	0.06	43.30	24.92	45.40
3	0.47	0.75	0.02	68.08	50.44	80.95
5	0.10	0.62	BDL	93.14	58.96	100
7	0.01	0.09	BDL	98.88	93.88	-
10	BDL	BDL	BDL	100	100	-

Fig. 1 Sample Collection

Fig. 2 Extraction

a. Extraction (Filteration) **b. Extraction (Separation)**

Fig. 3 Steps in estimation of insecticide residue

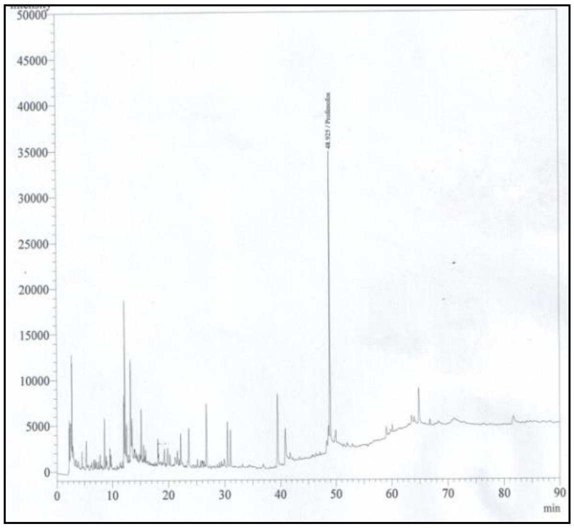

Fig. 4 Chromatograms of Prophenophos (400 g a.i/ha) Profenophos (7 DAS)

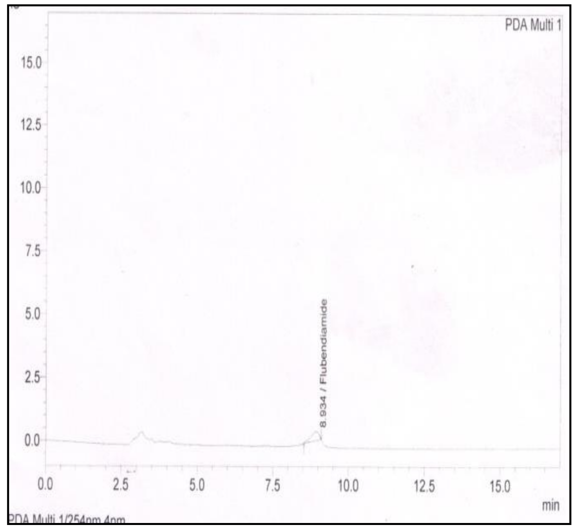

Fig. 5 Chromatograms of Flubendiamide (60 g a.i/ha) Flubendiamide (7 DAS)

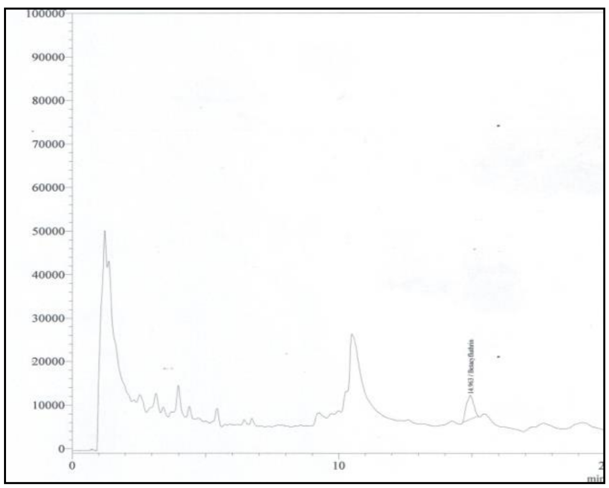

Fig. 6. Chromatograms of Beta-Cyfluthrin (18.75 g a.i/ha) Beta-cyfluthrin (3DAS)

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