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### Dissipation Pattern of Lambda Cyhalothrin on Chilli (Capsicum annum L.)

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### **ABSTRACT**

An experiment was conducted during kharif, 2015-16 to evaluate the efficacy of seven insecticides viz., fipronil 5% SC @ 500 g a.i ha<sup>-1</sup>, spinosad 45% SC @ 125 g a.i ha<sup>-1</sup>, chlorantraniliprole 20% SC @ 30 g a.i ha<sup>-1</sup>, profenophos 50% EC @ 400 g a.i ha<sup>-1</sup>, lambda cyhalothrin 5% SC @ 15.63 g a.i ha<sup>-1</sup>, imidacloprid + beta cyfluthrin 300% OD @ 30 g a.i ha<sup>-1</sup> and dimethoate 30 % EC @ 300 g a.i ha<sup>-1</sup> against chilli thrips. From the bio efficacy trail samples were collected at 0, 1, 3, 5, 7, 10 and 15 days after third spray during kharif 2015-16. Samples were analyzed at All India Network Project on Pesticide Residues, Rajendranagar, Hyderabad. The dissipation pattern of lambda cyhalothrin 5% SC @ 15.63 g a.i ha<sup>-1</sup> was studied collecting samples at regular intervals i.e. 0, 1, 3, 5, 7, 10 and 15days after last spray. The initial deposits of 1.20 mg kg<sup>-1</sup> lambda cyhalothrin recorded at 2 hours after last spray and dissipated to 0.78, 0.36 and 0.09 mg kg<sup>-1</sup> at 1, 3 and 5 days after last spray respectively and below determination level (BDL) by 7<sup>th</sup> day.

**Key words:** Insecticides, Thrips, Initial deposit, Efficacy, Dissipation, Below Determination Level.

### INTRODUCTION

Chilli (Capsicum annum L.), is an important vegetable and condiment crop grown throughout the world and it has immense commercial, dietary and therapeutiuc values. It is a rich source of A, C, E and P and an alkaloid capsacin, which has high medicinal value and is used in many pharmaceutical preparations. India is the world leader in chilli production followed by China and Pakistan.

The major chilli exporting countries with their percentage share in world exports are India (25%), China (24%), Spain (17%), Mexico (8%), Pakistan (7.2%), Morocco (7%) and Turkey (4.5%). The bulk share of chilli production in the world is held by Asian countries. In India chilli is cultivated in an area of 774.9 lakh ha with an annual production of 1492.1 lakh tones<sup>5</sup> (Horticultural Statistics, India 2015).

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Important chilli growing states in India are Andhra Pradesh, Telangana, Karnataka, Maharashtra and Tamilnadu which constitute nearly 75 per cent of the total area under chilli. Area under chilli crop in Andhra Pradesh and Telangana is around 1.72 lakh ha which is about 25.12 per cent of the total area in India. In Telangana State it is grown in 73,000 hectares with 2,53,000 tonnes production from major chilli growing areas such as Khammam, Warangal, Mahabubnagar and Ranga Reddy districts<sup>14</sup>.

Although the crop has great export potential besides huge domestic requirement, a number of limiting factors contribute for its low productivity. Among these various biotic stresses, ravages caused by insect pests are significant. The pest spectrum in chilli is complex with more than 293 insects and mite's species debilitating the crop in field as well as in storage<sup>2</sup>. Among these, chilli thrips, Scirtothrips dorsalis Hood has become the most notorious and pernicious pest on chilli. The overall reduction in fruit yield of chilli due to thrips and mites damage was up to 34 per cent<sup>13</sup>. These pests not only cause reduction in yield, but also act as vectors for several viral diseases and cause complete failure of crop and various biotic (pest and abiotic diseases), (rainfall, temperature, relative humidity and light intensity) and phenological factors (flower and fruit drop) limits the yield and quality of the chilli. A number of pesticides are being frequently used, to combat these pests. However, some of these insecticides leave residues on pods and

these residues may persist up to harvest. Presence of pesticide residues in the harvested chillies was posing problem at the time of export and in recent times importing countries have rejected few consignments. Pesticide use has increased rapidly over the last two decades at the rate of 12 per cent per year. The extensive and irrational use of pesticides resulted in the presence of residues of insecticides on chilli is likely to be associated with severe effects on human health. Hence, great significance has to be given to estimate pesticide residues in chilli.

### MATERIALS AND METHODS

The experiment was laid out in a Randomized Block Design (RBD) with 8 treatments including untreated control replicated thrice with individual plot size of 20 m<sup>2</sup> (5mx4 m) and the insecticides viz., fipronil 5% SC @ 500 g a.i ha<sup>-1</sup>, spinosad 45% SC @ 125 g a.i ha<sup>-1</sup>, chlorantraniliprole 20% SC @ 30 g a.i ha<sup>-1</sup> <sup>1</sup>, profenophos 50% EC @ 400 g a.i ha<sup>-1</sup>, lambda cyhalothrin 5% SC @ 15.63 g a.i ha<sup>-1</sup>, imidacloprid + beta cyfluthrin 300% OD @ 30 g a.i ha<sup>-1</sup> and dimethoate 30 % EC @ 300 g a.i ha<sup>-1</sup> on chilli first at 50% flowering and the second and third spray ten days later to evaluate the efficacy against thrips. and the dissipation studies were conducted for the same by collecting chilli samples at regular intervals i.e. 0, 1, 3, 5, 7, 10 and 15 days after last spray in polythene bags and brought to the laboratory immediately for further sample processing in the laboratory as detailed here under.

### Extraction and Clean -Up

The sample was homogenized at 14000-15000 rpm for 2-3 min using Heidolph silent crusher

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3±0.1g sodium chloride was added to tube and mixed by shaking gently

Centrifuged for 3 min at 2500-3000 rpm to separate the organic layer

The top organic layer of about 16 ml was taken into the 50 ml centrifuge tube

9±0.1 g anhydrous sodium sulphate was added to remove the moisture content

8 ml of extract was taken in to 15 ml tube containing  $0.4{\pm}0.01g$  PSA sorbent (for dispersive solid phase d-SPE cleanup) and

1.2±0.01 gr anhydrous magnesium sulphate

The sample tube was vertexed for 30 sec followed by centrifugation for 5 min at 2500-3000 rpm

The extract of about 2ml was transferred into test tubes and evaporated to dryness using turbovap with nitrogen gas and reconstituted with 1ml n-Hexane: Acetone (9:1) for GC analysis with ECD for lambda cyhalothrin analysis.

### **Gas Chromatograph parameters**

| Gas Chromatograph                        | Gas Chromatography- AGILENT- 7890B                         |  |  |  |  |
|--|--|--|--|--|--|
| Column                                   | VF -5ms Capillary Column                                   |  |  |  |  |
|  | 30 m length, 0.25 mm Internal Diameter, 0.25 □m film       |  |  |  |  |
|  | thickness; 1% methyl siloxane                              |  |  |  |  |
| Column Oven ( <sup>0</sup> C)            | Initial 200°C for 6 min - increase @ 20°C/min upto 280°C - |  |  |  |  |
|  | hold for 10 mins   |  |  |  |  |
| Detectors                                | Electron Capture Detector (ECD)                            |  |  |  |  |
| Detector Temperature ( <sup>0</sup> C)   | 300  |  |  |  |  |
| Injector Temperature ( <sup>0</sup> C)   | 280  |  |  |  |  |
| Injector Status                          | Split Ratio: 1:2   |  |  |  |  |
| Carrier Gas                              | Nitrogen, Iolar II, Purity 99.999%                         |  |  |  |  |
| Carrier Gas Flow (ml min <sup>-1</sup> ) | 2  |  |  |  |  |
| Make-up Flow (ml min <sup>-1</sup> )     | 25   |  |  |  |  |
| Retention time (min)                     | 9.11   |  |  |  |  |
| Total run time (min)                     | 20   |  |  |  |  |

## Fortication Anf Recovery Results of Lambda Cyhalothrin On Chilli

Lamda-Cyhalothrin at 0.05 Mg Kg<sup>-1</sup>, 0.25 Mg Kg<sup>-1</sup> And 0.5 Mg Kg<sup>-1</sup> Were Analysed and The Mean Recovery Of The Residues Using The Method Was 88.10, 86.67 And 84.56 Per Cent,

Respectively In Green Chilli. The Results Shown That The Method Was Suitable For The Analysis Of Lamda Cyhalothrin Residues Up To 0.05 Mg Kg<sup>-1</sup>, And The Limit Of Quantification (LOQ) Was 0.05 Mg Kg<sup>-1</sup>.

### Recovery of lambda-cyhalothrin from fortified green chilli samples

| Details | Recoveries of lambda-cyhalothrin from fortified chilli samples |            |   |            |   |            |  |  |
|---------|--|------------|---|------------|---|------------|--|--|
|         | Fortified level (mg kg <sup>-1</sup> )                         |            |   |            |   |            |  |  |
|         | <b>0.05</b> mg kg <sup>-1</sup>                                |            | <b>0.25</b> mg kg <sup>-1</sup>                 |            | <b>0.5</b> mg kg <sup>-1</sup>                  |            |  |  |
|         | Residues<br>recovered<br>(mg kg <sup>-1</sup> )                | Recovery % | Residues<br>recovered<br>(mg kg <sup>-1</sup> ) | Recovery % | Residues<br>recovered<br>(mg kg <sup>-1</sup> ) | Recovery % |  |  |
| R1      | 0.046  | 91.74      | 0.217   | 86.86      | 0.412   | 82.50      |  |  |
| R2      | 0.044  | 84.46      | 0.216   | 86.55      | 0.433   | 86.61      |  |  |
| R3      | 0.042  | 84.78      | 0.216   | 86.60      | 0.422   | 84.15      |  |  |
| Mean    |  | 88.10      |   | 86.67      |   | 84.56      |  |  |
| SD      |  | 5.14       |   | 0.16       |   | 2.91       |  |  |
| RSD     |  | 5.84       |   | 0.19       |   | 3.44       |  |  |

Hence, the method described above is suitable for the analysis of samples collected from the field sprayed with lambda cyhalothrin residues to study the residue dynamics / dissipation pattern. Samples of chilli were collected from lambda cyhalothrin 5% SC @ 15.63 g a.i ha<sup>-1</sup>

sprayed plots at regular intervals i.e. 0, 1, 3, 5, 7, 10 and 15 days after last spray and analyzed for residues following the validated methods. Residues (mg kg<sup>-1</sup>) were calculated using the formula given below.

The following parameters were calculated to know the dissipation pattern of the insecticides on chilli. Dissipation per centage:

$$\begin{tabular}{ll} Initial deposit - Residues at given time \\ \hline Per cent dissipation = & & & & & X 100 \\ \hline & & & & & & Initial deposit \\ \hline \end{tabular}$$

Waiting period: Waiting period  $(T_{tol})$  is defined as the minimum number of days to lapse before the insecticide reaches the

tolerance limit. The waiting periods were calculated by the following formula.

Ttol = 
$$\frac{[a - \text{Log tol }]}{b}$$

Where

T<sub>tol</sub> = Minimum time (in days) required for the pesticide residue to reach below the tolerance limit.

a = Log of apparent initial deposits obtained in the regression equation

(Y = a+bX)

tol = Tolerance limit of the insecticide (MRL)

b = Slope of the regression line

### RESULTS AND DISCUSSION

Lambda cyhalothrin was sprayed thrice @ 15.63 g a.i. ha<sup>-1</sup> on chilli and green chilli samples were collected at regular intervals of 0, 1, 3, 5, 7, 10 and 15 days after third spray. The samples were processed and estimated for the residues of lambda cyhalothrin on Gas Chromatograph (GC - ECD). The results showed that the initial deposits of 1.20 mg kg<sup>-1</sup> were detected in green chilli. The residues recorded at 1, 3 and 5 days after third spraying were found to be 0.78, 0.36 and 0.09 mg kg<sup>-1</sup>, showing a dissipation per cent of 35.00, 70.00 and 92.50, respectively at 7 days after last spray. The residues were below detectable level (BDL) showing 100 per cent dissipation.

Based on the dissipation curve waiting periods have been worked out using linear semi-logarithmic regression analysis<sup>6</sup>. The results showed that the residues of lambda cyhalothrin reached to below tolerance limit of 0.05 mg kg<sup>-1</sup> (As per FSSAI) in 11.16 days. The regression equation was Y = 1.089 + (-0.214) X with  $R^2 = 0.952$ .

Sharma and Awasthi<sup>11</sup> (2002) reported that average initial deposits of lambda cyhalothrin (2.5 EC and 5.0 EC) on cauliflower found to be 0.81 to 1.59 mg kg<sup>-1</sup> following application at 15 and 30 g a.i. ha<sup>-1</sup>. Residues of lambda cyhalothrin (0.81 to 1.59 mg kg<sup>-1</sup>) dissipated to below detectable level by 10 days at the single dose and 15 days from the double dose. The waiting periods were between 4.2 to 5.2 days from different

treatments, while Ahuja *et al*<sup>1</sup>., sprayed lambda cyhalothrin on brinjal at both recommended (@ 15 g a.i. ha<sup>-1</sup>) and double dosage (@ 30 g a.i. ha<sup>-1</sup>) and found that the initial deposits of 0.75 mg kg<sup>-1</sup>, and 1.27 mg kg<sup>-1</sup> dissipated to BDL in 10 days, respectively. The variation of results from the present findings may be due to change in matrix, dosage and time of application.

The present findings differ from Reddy et al10., using lambda cyhalothrin on chilli at 50 g a.i. ha<sup>-1</sup>, recorded initial deposits of 0.62 mg kg<sup>-1</sup>. Further, it was also reported that the initial deposits may vary with formulation for the same dosage. Mahmoud and Soliman<sup>8</sup> who reported that an average initial deposit of 8.76 ppm of lambda cyhalothrin on chickpea, which reached below determination level by 15th day. Elbashir et  $al^3$ ., found the initial residues of 3.04 ppm, when lambda cyhalothrin was applied at 30 g a.i. ha<sup>-1</sup> in tomato and suggested safe waiting period of 18 days. Gupta et al<sup>4</sup>., reported that lambda cyhalothrin applied at 15 g a.i. ha<sup>-1</sup> recorded initial deposits of 0.138 mg kg<sup>-1</sup>, dissipated to 92.75 per cent on 10<sup>th</sup> day after application on brinjal. The initial deposits and dissipation vary from crop to crop depending up on the crop canopy, season, age of the crop, sample matrix, surface area of sample etc<sup>7</sup>., and the same can be witnessed based on the test reports published by Pawar and jadhav<sup>9</sup>, Singh and Singh<sup>12</sup> and Ahuja et al<sup>1</sup>., on various crops at different doses.

Table 1: Dissipation pattern of lambda cyhalothrin5% SC (15.63 g a.i ha<sup>-1</sup>) in chilli after three sprays

|  | Residues               |      |      |         |               |  |  |
|--|------------------------|------|------|---------|---------------|--|--|
| Days after last spray                  | R1                     | R2   | R3   | Average | Dissipation % |  |  |
| 0                                      | 1.19                   | 1.17 | 1.23 | 1.20    |               |  |  |
| 1                                      | 0.78                   | 0.77 | 0.78 | 0.78    | 35.00         |  |  |
| 3                                      | 0.32                   | 0.35 | 0.43 | 0.36    | 70.00         |  |  |
| 5                                      | 0.10                   | 0.09 | 0.09 | 0.09    | 92.50         |  |  |
| 7                                      | BDL                    | BDL  | BDL  | BDL     | 100.00        |  |  |
| 10                                     | BDL                    | BDL  | BDL  | BDL     | 100.00        |  |  |
| 15                                     | BDL                    | BDL  | BDL  | BDL     | 100.00        |  |  |
| Regression equation                    | Y = 1.089 + (-0.214) X |      |      |         |               |  |  |
| $\mathbb{R}^2$                         | 0.952                  |      |      |         |               |  |  |
| MRL (As per FSSAI) mg kg <sup>-1</sup> | 0.05                   |      |      |         |               |  |  |
| Waiting period (days)                  | 11.16                  |      |      |         |               |  |  |

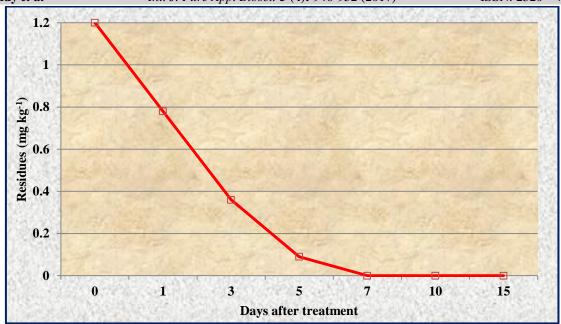


Fig. 1: Dissipation kinetics of lambda cyhalothrin residues in chilli after three sprays

### CONCLUSION

The initial deposits and waiting period for safe harvest of chilli when sprayed thrice with lambda cyhalothrin 5% SC @ 15.63 g a.i ha<sup>-1</sup> were 1.20 mg kg<sup>-1</sup> and 11.16 days, respectively.

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