

## Dissipation Pattern of Chlorantraniliprole 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 chlorantraniliprole 20% SC @ 30 g a.i ha<sup>-1</sup> was studied collecting samples at regular intervals i.e. 0, 1, 3, 5, 7, 10 and 15 days after last spray. The initial deposits of 0.56 mg kg<sup>-1</sup> chlorantraniliprole recorded at 2 hours after last spray and dissipated to 0.31, 0.17 and 0.06 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 therapeutic values. It is a rich source of A, C, E and P and an alkaloid capsaicin, 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>3</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>6</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 mites species debilitating the crop in field as well as in storage<sup>1</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>5</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 diseases), abiotic (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

#### Extraction and Clean –Up

Chilli fruits ( 5kg) were homogenized with robot coupe blixer and homogenized

↓  
15±0.1g sample was taken in 50 ml centrifuge tube

↓  
Required quantity of standard (CRM) added to get desired fortification level

↓  
30±0.1 ml acetonitrile was added to the tube

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



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>, 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.

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±0.01g PSA sorbent (for dispersive solid phase d-SPE cleanup) and

1.2±0.01 gr anhydrous magnesium sulphate



The sample tube was vortexed 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 chlorantraniliprole 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 µm film thickness; 1% methyl siloxane
Column Oven (°C)	Initial 180°C for 2 min - increase @ 10°C/min upto 260°C – hold for 15 mins.
Detectors	Electron Capture Detector (ECD)
Detector Temperature (°C)	300
Injector Temperature (°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)	4.18
Total run time (min)	24

### Fortication and Recovery results of chlorantraniliprole on chilli

Chilli samples fortified with chlorantraniliprole at 0.05 mg kg<sup>-1</sup>, 0.25 mg kg<sup>-1</sup> and 0.5 mg kg<sup>-1</sup>, respectively were analysed and the mean recovery of the

residues using the method was 99.23, 94.68 and 88.27 per cent, respectively in green chilli. The results shown that the method was suitable for the analysis of chlorantraniliprole 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 chlorantraniliprole from fortified green chilli samples

Details	Recovery of chlorantraniliprole from fortified chilli samples					
	Fortified level (mg kg <sup>-1</sup> )					
	0.05 mg kg <sup>-1</sup>		0.25 mg kg <sup>-1</sup>		0.50 mg kg <sup>-1</sup>	
	Residues recovered (mg kg <sup>-1</sup> )	Recovery %	Residues recovered (mg kg <sup>-1</sup> )	Recovery %	Residues recovered (mg kg <sup>-1</sup> )	Recovery %
R1	0.050	100.80	0.244	97.71	0.446	89.15
R2	0.053	105.20	0.232	92.78	0.433	86.68
R3	0.046	91.70	0.234	93.55	0.445	89.00
Mean		99.23		94.68		88.27
SD		6.907		2.652		1.379
RSD		6.961		2.801		1.563

Hence, the method described above is suitable for the analysis of samples collected from the field sprayed with chlorantraniliprole residues to study the residue dynamics / dissipation pattern. Samples of chilli were collected from chlorantraniliprole 20% SC @ 30 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 analysed for residues following the validated methods. Residues (mg kg<sup>-1</sup>) were calculated using the formula given below.

$$\text{Residues (mg kg}^{-1}\text{)} = \frac{\text{Sample peak area X conc of std (ppm) X } \mu\text{l std. injected X Final volume of the sample (2 ml)}}{\text{Standard Peak area X weight of sample analysed (2 g) X } \mu\text{l of sample injected}} \times \text{recovery factor}$$

The following parameters were calculated to know the dissipation pattern of the insecticides on chilli.

**Dissipation per centage:**

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

**Waiting period:** Waiting period (T<sub>tol</sub>) 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.

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

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

Chlorantraniliprole was sprayed thrice @ 30 g a.i. ha<sup>-1</sup> viz., first spray was given at 50 per cent flowering while second and third spray at 10 days after each spray. The green chilli samples were collected after third spray at regular intervals of 0, 1, 3, 5, 7, 10 and 15 days. The samples were processed and estimated on gas chromatograph (GC - ECD) for chlorantraniliprole. The results showed that the initial deposits of 0.56 mg kg<sup>-1</sup> were dissipated to 0.06 mg kg<sup>-1</sup> by 5<sup>th</sup> day after third spray on green chilli. The residues of 0.31, 0.17 and 0.06 mg kg<sup>-1</sup> were recorded at 1, 3 and 5 days after last spray, respectively. However, residues were below detectable level (BDL) and showed 100.00 per cent dissipation from 7 days after third spray.

Based on the first order kinetics, waiting periods have been worked out using linear semi-logarithmic regression analysis<sup>2</sup>.

Dissipation pattern showed a continuous decrease of residues from 1<sup>st</sup> day to 7<sup>th</sup> day. The residues dissipated to 44.64, 69.64, 89.26 and 100.00 per cent on 1, 3, 5 and 7 days, respectively. The initial deposit of chlorantraniliprole to reach below tolerance limit (T<sub>tol</sub>) of 0.03 mg kg<sup>-1</sup> (As per FSSAI) was 21.98 days. The regression equation was  $Y = 0.481 + (-0.091) X$  with  $R^2 = 0.891$ .

Kar *et al*<sup>4</sup>. (2013) reported that the initial deposits of chlorantraniliprole 18.5 SC on cauliflower from treatments @ 9.25 and 18.50 g a.i. ha<sup>-1</sup> were 0.18 and 0.29 mg kg<sup>-1</sup>, respectively and waiting period of 1 day was suggested for safe consumption of cauliflower curds. 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.

**Table 1: Dissipation pattern of chlorantraniliprole 20% SC (30 g a.i ha<sup>-1</sup>) in chilli after three sprays**

Days after last spray	Residues of chlorantraniliprole (mg kg <sup>-1</sup> )				Dissipation %
	R1	R2	R3	Average	
0	0.54	0.59	0.55	0.56	--
1	0.33	0.28	0.31	0.31	44.64
3	0.18	0.18	0.16	0.17	69.64
5	0.07	0.06	0.06	0.06	89.28
7	BDL	BDL	BDL	BDL	100.00
10	BDL	BDL	BDL	BDL	100.00
15	BDL	BDL	BDL	BDL	100.00
Regression equation	$Y = 0.481 + (-0.091) X$				
R <sup>2</sup>	0.891				
MRL (As per FSSAI) mg kg <sup>-1</sup>	0.03				
Waiting period (days)	21.98				

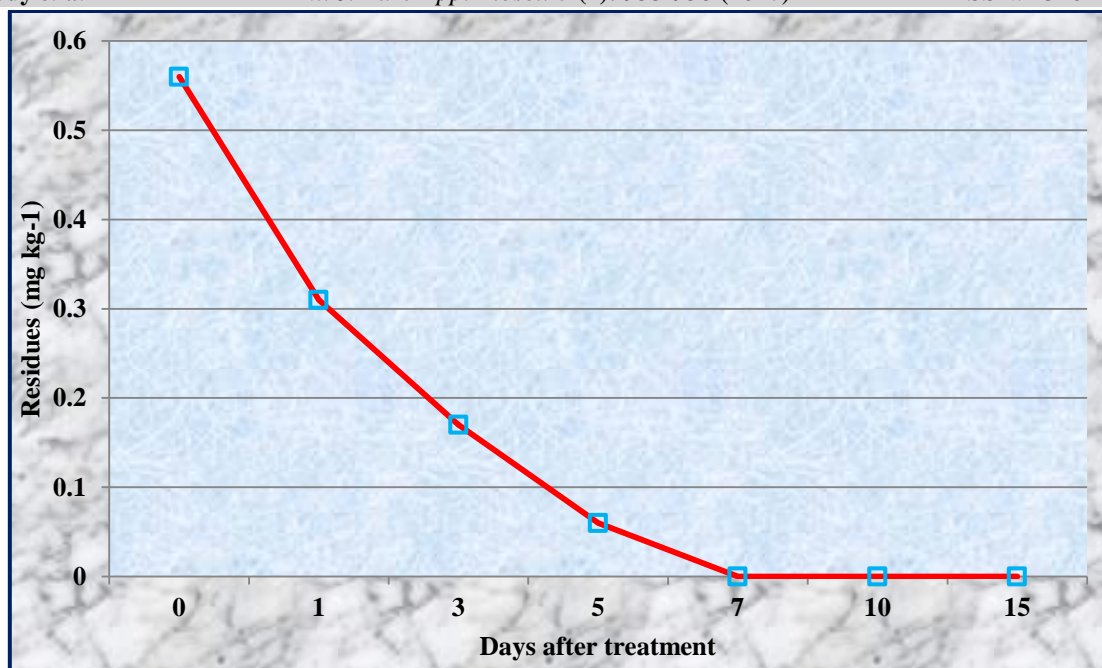


Fig. 1: Dissipation kinetics of chlorantraniliprole residues in chilli after three Spray

### CONCLUSION

The initial deposits and waiting period for safe harvest of chilli when sprayed thrice with chlorantraniliprole at 30 g a.i. ha<sup>-1</sup> were 0.56 mg kg<sup>-1</sup> and 21.98 days, respectively.

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