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Dissipation pattern of spinosad on Chilli (*Capsicum annum* L)

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Abstract
An experiment was conducted during *kharif*, 2015 to evaluate the efficacy of seven insecticides *viz.*, fipronil 5% SC @ 500 g a.i ha^{-1}, spinosad 45% SC @ 125 g a.i ha^{-1}, chlorantraniliprole 20% SC @ 30 g a.i ha^{-1}, profenophos 50% EC @ 400 g a.i ha^{-1}, lambda cyhalothrin 5% SC @ 15.63 g a.i ha^{-1}, imidacloprid + beta cyfluthrin 300% OD @ 30 g a.i ha^{-1} and dimethoate 30 % EC @ 300 g a.i ha^{-1} against chilli thrips. The dissipation pattern of spinosad 45% SC @ 125 g a.i ha^{-1} was studied collecting samples at regular intervals *i.e.* 0, 1, 3, 5, 7, 10 and 15 days after last spray and analyzed. The initial deposits of 0.78 mg kg^{-1} were dissipated to 0.10 mg kg^{-1} by 5th day after third spray on chilli. The residues of 0.43, 0.18 and 0.10 mg kg^{-1} were recorded at 1, 3 and 5 days after last spray, respectively. However residues were below detectable level (BDL) from 7 days after third spray and showed 100.00 per cent dissipation.

Keywords: Insecticides, thrips, initial deposit, spinosad, efficacy and dissipation

1. 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 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 (Horticultural Statistics, India 2015) [2]. 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 (*WWW. Indiastat.com*) [9].

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 (Butani, 1976) [1]. 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 (Thania *et al.*, 2011) [7]. 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 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.

2. 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^2 (5mx4 m) and the insecticides *viz.*, fipronil 5% SC @ 500 g a.i ha^{-1}, spinosad 45% SC @ 125 g a.i ha^{-1}, chlorantraniliprole 20% SC @ 30 g a.i ha^{-1}, profenophos 50% EC @ 400 g a.i ha^{-1}, lambda cyhalothrin 5% SC @ 15.63 g a.i ha^{-1}, imidacloprid + beta cyfluthrin 300% OD @ 30 g a.i ha^{-1} and dimethoate 30 % EC @ 300 g a.i ha^{-1} 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 cabbage 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

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

↓

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 vertexed for 30 sec followed by centrifugation for 5 min at 2500-3000 rpm

↓

For analysis of spinosad, 2 ml extract was filtered by using PTFE filter and finally 1 ml filtered extract is taken for injection in to the HPLC with Mass Spectrometer (MS)

Table 1: Details of HPLC operating parameters

HPLC	**SHIMADZU LC-20**		
Detector	Mass Spectrometer (MS)		
Column	HPLC Column Kinetex C18 column, 2.6 micron particle size 100 length, 3 mm ID		
Solvents in Pump A	Water		
Solvents in Pump B	Metanol		
Solvents Gradient Program	Water: Methanol (5:95) mixture run for 2 min		
Solvents Gradient rate	0.4 ml min^{-1}		
Quantity of sample injected	1 µl		
Run time	10 min		
Retention time	Spinosad – 2.70 min		
LC Program For spinosad	Time	Methanol	Water
	0.01	95	5
	3.00	85	15
	5.00	95	5
	5.01	Stop	

Fortication and Recovery results of spinosad on chilli

Chilli samples fortified with spinosad at 0.05 mg kg^{-1}, 0.25 mg kg^{-1} and 0.5 mg kg^{-1}, respectively were analysed and the mean recovery of the residues using the method was 93.48, 96.88 and 102.32 per cent, respectively in green chilli (Table 1). The results shown that the method was suitable for the analysis of spinosad residues up to 0.05 mg kg^{-1} and the limit of quantification (LOQ) was 0.05 mg kg^{-1}.

Table 2: Recovery of spinosad from fortified green chilli samples

Details	Recoveries of spinosad from fortified chilli samples					
	Fortified level (mg kg^{-1})					
	0.05 mg kg^{-1}		0.25 mg kg^{-1}		0.50 mg kg^{-1}	
	Residues recovered (mg kg^{-1})	Recovery %	Residues recovered (mg kg^{-1})	Recovery %	Residues recovered (mg kg^{-1})	Recovery %
R1	0.046	91.63	0.248	99.01	0.504	100.84
R2	0.047	94.91	0.237	94.66	0.509	101.83
R3	0.047	93.89	0.242	96.96	0.512	104.30
Mean		93.48		96.88		102.32
SD		1.67		2.174		1.780
RSD		1.78		2.24		1.74

Hence, the method described above is suitable for the analysis of samples collected from the field samples sprayed with spinosad to study the dissipation pattern in green chilli**.** Residues (mg kg^{-1}) were calculated using the formula given below.

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

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

Dissipation percentage

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

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_{tol} = 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

3. Results and Discussion

Spinosad was sprayed thrice @ 125 g a.i. ha^{-1} with first spray at 50 per cent flowering while second and third spray at 10 days after each spray. The green chilli samples were collected at regular intervals of 0, 1, 3, 5, 7, 10, and 15 days after third spray. The samples were estimated for residues of spinosad on High Performance Liquid Chromatograph (HPLC) after processing. The initial deposits of 0.78 mg kg^{-1} were dissipated to 0.10 mg kg^{-1} by 5th day after third spray on chilli. The residues of 0.43, 0.18 and 0.10 mg kg^{-1} were recorded at 1, 3 and 5 days after last spray, respectively. However residues were below detectable level (BDL) from 7 days after third spray and showed 100.00 per cent dissipation. The dissipation pattern of spinosad was presented in table 2 and depicted in figure. 1.

Using linear semi-logarithmic regression analysis and based on the first order kinetics, waiting period was worked out (Hoskins, 1961) [3]. There was continuous decrease of residues from 1st to 7th day. The residues dissipated to 44.87, 76.92, 98.72 and 100.00 per cent on 1, 3, 5 and 7 days, respectively. The initial deposit of spinosad to reach below tolerance limit (T_{tol}) of 0.001 mg kg^{-1} (as per FSSAI) was 28.81 days. The regression equation was Y= 0.659 + (-0.127) X with R^2 = 0.856.

Mandal *et al.* (2009) [5] found, the initial deposits of spinosad were 0.57 and 1.34 µg kg^{-1}, respectively following three sprays of spinosad 2.5 SC at 15 and 30 g a.i. ha^{-1}, respectively on cauliflower. Residues of spinosad dissipated to below the limit of quantification (LOQ) of 0.02 µg kg^{-1} after 10 days at both the doses and waiting period of six days was suggested, while Singh *et al.* (2012) [6] reported the initial deposits as 0.33 and 0.56 µg kg^{-1} of spinosad at single and double doses, respectively *viz.*,15 and 30 g a.i. ha^{-1}.They dissipated below its limit of quantification of 0.01 µg kg $^{-1}$ after five and seven days at single and double doses, respectively on cabbage. Vijayasree *et al.* (2014) [8] reported the initial deposits of 0.94 and 1.9 µg kg^{-1} of spinosad reached below detectable level on the seventh day and fifteenth day at 73 and 146 g a.i. ha^{-1}, respectively and calculated safe waiting period of 1.09 – 3.25 days on cowpea. The slight variation in initial deposits from the present findings may be due to variation in matrix and dosage of the insecticide applied (Khay *et al.*, 2008) [4].

Table 3: Dissipation pattern of spinosad 45% SC (125 g a.i ha^{-1}) in chilli after three sprays

Days after last spray	Residues of spinosad (mg kg^{-1})				Dissipation %
	R1	R2	R3	Average	
0	0.77	0.82	0.74	0.78	--
1	0.47	0.45	0.39	0.43	44.87
3	0.16	0.19	0.19	0.18	76.92
5	0.10	0.11	0.10	0.10	98.72
7	BDL	BDL	BDL	BDL	100.00
10	BDL	BDL	BDL	BDL	100.00
15	BDL	BDL	BDL	BDL	100.00
Regression equation	Y = 0.659 + (-0.127) X				
R^2	0.856				
MRL (As per FSSAI) mg kg^{-1}	0.001				
Waiting period (days)	28.81				

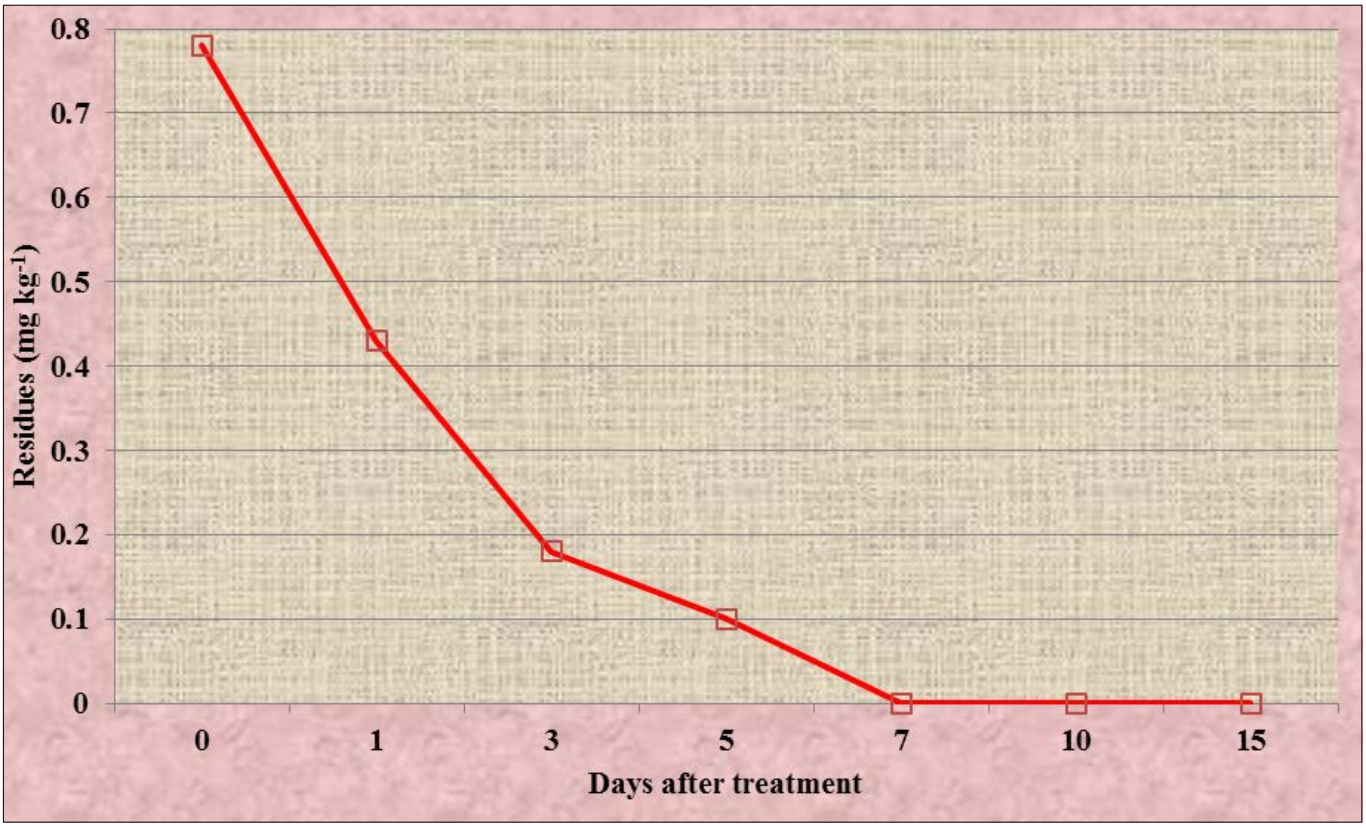

Fig 1: Dissipation kinetics of spinosad residues in chill after three sprays

4. Conclusion

From this experiment, it can be concluded that initial deposits of spinosad 45% SC (125 g a.i ha-1) after three sprays were 0.78 mg kg^{-1} after third spray in chilli which dissipated to below detectable level at 7 days. The waiting period determined for spinosad 45% SC (125 g a.i ha-1) after third spray in chilli was 28.81 days.

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