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Dissipation Pattern of Dimethoate 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 dimethoate 30 % EC @ 300 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 3.86 mg kg^{-1} dimethoate recorded at 2 hours after last spray dissipated to 2.66, 2.28, 1.06, 0.22 and 0.08 mg kg^{-1} at 1, 3, 5, 7 and 10 days, after last spray respectively and below determination level (BDL) by 15th day.

Key words: *Insecticides, Thrips, Initial Deposit, Dimethoate, Efficacy, Dissipation.*

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 (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.

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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[10].

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)[2]. 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.

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

↓

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 dimethoate 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 (^{0}C)	Dimethoate - Initial 150^{0}C for 1 min - increase @ 20^{0}C/min upto 250^{0}C – hold for 14 mins.
Detectors	Electron Capture Detector (ECD)
Detector Temperature (^{0}C)	300
Injector Temperature (^{0}C)	280
Injector Status	Split Ratio: 1:2
Carrier Gas	Nitrogen, Iolar II, Purity 99.999%
Carrier Gas Flow (ml min^{-1})	2
Make-up Flow (ml min^{-1})	25
Retention time (min)	Dimethoate 6.12
Total run time (min)	Dimethoate 20
Gas Chromatograph	Gas Chromatography-VARIAN GC 3800
Column	VF-1ms Capillary Column 30 m length, 0.25 mm Internal Diameter, 0.25 µm film thickness; 1% methyl siloxane
Column Oven (^{0}C)	240 (Isothermal)
Detectors	Electron Capture Detector (ECD) Thrmionic Specific Detector (TSD)
Detector Temperature (^{0}C)	280
Injector Temperature (^{0}C)	260
Injector Status	Front Injector Type 1177 Split / Splitless Split ratio: 10
Carrier Gas	Nitrogen, Iolar II, Purity 99.99%
Carrier Gas Flow (ml min^{-1})	1 ml/min
Make-up Flow (ml min^{-1})	35 ml/min
Retention time (min)	Dimethoate min
Total run time (min)	30 min

Fortication ANF Recovery results of dimethoate on chilli

Chilli samples fortified with dimethoate at 0.05 mg kg^{-1}, 0.25 mg kg^{-1} and 0.5 mg kg^{-1} were analysed and the mean recovery of the residues using the method was 88.45, 106.57 and 103.96 per cent, respectively in green chilli. The results shown that the method was suitable for the analysis of dimethoate residues up to 0.05 mg kg^{-1}, and the limit of quantification (LOQ) was 0.05 mg kg.

Table 3.14. Recovery of dimethoate from fortified green chilli samples

Details	**Recoveries of dimethoate from fortified chilli samples**					
	Fortified level (mg kg^{-1}**)**					
	0.05 mg kg^{-1}		**0.25** mg kg^{-1}		**0.5** mg kg^{-1}	
	Residues recovered (mg kg^{-1}**)**	**Recovery %**	**Residues recovered (**mg kg^{-1}**)**	**Recovery %**	**Residues recovered (**mg kg^{-1}**)**	**Recovery %**
R1	**0.042**	85.70	**0.265**	106.18	**0.519**	103.89
R2	**0.045**	90.21	**0.268**	107.43	**0.512**	102.58
R3	**0.047**	89.45	**0.265**	106.09	**0.527**	105.41
Mean		88.45		106.57		103.96
SD		2.41		0.75		1.42
RSD		2.73		0.70		1.36

Hence, the method described above is suitable for the analysis of samples collected from the field sprayed with profenophos residues to study the residue dynamics / dissipation pattern. Samples of chilli were collected from profenophos @ 400 g a.i./ha 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^{-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 cabbage.

Dissipation per centage:

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

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.

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

Where

T_{tol} = Minimum time (in days) required for the pesticide residue to reach below the tolerance limit.

a = 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

Dimethoate was sprayed thrice @ 30 g a.i. ha^{-1} and green chilli samples were collected at regular intervals of 0, 1, 3, 5, 7, 10 and 15 days after last spray. The samples were processed and estimated for residues of dimethoate on Gas Chromatograph (GC - ECD). The dissipation pattern was presented in table 4.23 and depicted in figure 4.11. The results showed that the initial deposits of 3.86 mg kg^{-1} were detected in chilli. The residues recorded at 1, 3, 5, 7 and 10 days after third spraying were found to be 2.66, 2.28, 1.06, 0.22 and 0.08 mg kg^{-1}, showing a dissipation per cent of 31.09, 40.93, 72.54, 94.30 and 97.93, respectively. After 15 days, 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. The results showed that the residues of dimethoate reached to below tolerance limit of 0.50 mg kg^{-1} in 6.64 days. The regression equation was Y= 3.326 + (-0.376) X with R^2 = 0.909.

The results were in agreement with the findings of Sharma and Parihar[5] (2013) who reported that an average initial deposit of 3.12 and 5.16 mg kg^{-1}, respectively of dimethoate was observed when sprayed at 300 and 600 g a.i.ha^{-1} on chilli with waiting periods were 3.29 and 4.50 days, while Khan[3] (1997) reported that the initial deposit of dimethoate 30 EC were found to be 2.93 ppm on okra fruits which reached to safe waiting period of 3.17 days. Soudamini *et al.*[6] (2004) also reported that the initial deposits of dimethoate 0.05% and 0.10% were 2.10 and 3.50 mg kg^{-1}, respectively on acid lime. The variation in the waiting period in the present finding may be due to variation in the matrix.

The present findings differ from Waghulde *et al.*[9] (2011) who reported that an initial deposit of 8.01 ppm in chilli and 22.90 ppm of dimethoate 30 EC was observed in okra when sprayed at 6 g a.i. ha^{-1} and also Varghese *et al* .[8] *(*2012) observed initial deposit of 7.36 ppm on chilli after application at 300 g a.i.ha^{-1}. In another study Reddy *et al.*[4] (2007) recorded the initial deposit of dimethoate in chilli was 0.33 ppm when applied at 300 g a.i. ha^{-1}, which dissipated to non detectable level at 15 days after application and they suggested safe waiting period of one day. This variation may be due to change in the matrix of the different crops. 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 dimethoate 30% EC (300 g a.i ha^{-1}) in chilli after three sprays

Days after last spray	Residues of dimethoate (mg kg^{-1})				Dissipation %
	R1	R2	R3	Average	
0	3.82	3.96	3.78	3.86	0
1	2.75	2.68	2.55	2.66	31.09
3	2.24	2.28	2.31	2.28	40.93
5	1.12	1.10	0.95	1.06	72.54
7	0.22	0.22	0.23	0.22	94.30
10	0.08	0.08	0.08	0.08	97.93
15	BDL	BDL	BDL	BDL	100
Regression equation	Y = 3.326 + (-0.376) X				
R^2	0.909				
MRL (As per FSSAI) mg kg^{-1}	0.50				
Waiting period (days)	6.64				

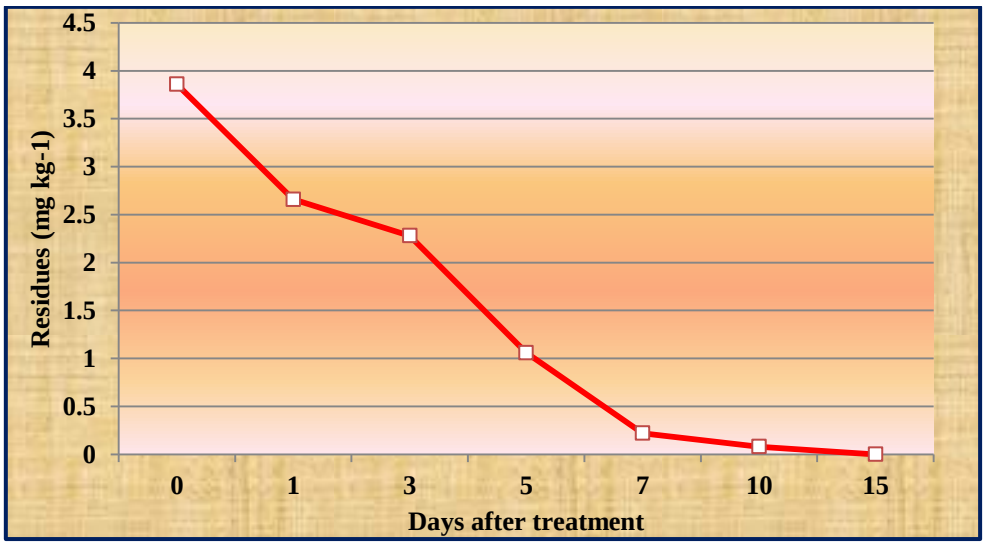

Fig. 1: Dissipation kinetics of dimethoate residues in chilli after three sprays

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