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Persistence studies of Deltamethrin 2.8% in chickpea (*Cicer arietinum* Linn.) and soil in southern Telangana region

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Abstract

Dissipation behavior of deltamethrin 2.5% EC (12.5 and 25 g a.i./ha) when applied on chickpea (var. Jaki 1211) thrice at 7 days interval for controlling pod borer under field conditions during *rabi* 2015-16 at pod initiation stage was studied. Green pods from recommended dose of deltamethrin retained residues (0.059 mg/kg) upto 7 days whereas from double the recommended dose retained residues (0.082 mg/kg) upto 10 days after final spray and dissipated to below limit of quantitation of 0.05 mg/kg by 10th and 15th day with half-life of 1.123 and 1.135 days respectively. Mature pods and soil at harvest contained residues below limit of quantitation (0.05 mg/kg).

Keywords: Deltamethrin, dissipation, persistence, residues, chickpea pods, soil

Introduction

Chickpea (*Cicer arietinum* L.) is one of the most important pulse crops which is cultivated worldwide. India is the world's largest producer and consumer of chickpea which occupies first position in the world in area and production. In India, the crop is grown in 8.25 m ha area with the production of 7.33 Mt and productivity of 889 kg ha⁻¹ whereas, in Telangana, chickpea is cultivated in about 70,000 ha with production of 49,000 tonnes with the productivity of 700 kg ha⁻¹ (Agricultural Statistics at a Glance, 2018). It has been reported that losses caused by the biotic constraints, which include insect pests, diseases and weeds under field conditions are the main reasons for decline in the area, production and productivity for the last few decades. About 36 different species of insect pests has been reported to attack chickpea during different growth stages of the crop in India (Nayer *et al.*, 1982) [7] of which gram pod borer (*Helicoverpa armigera* Hubner) is the only major and most devastating pest which alone causes 29% yield losses in chickpea at national level. It is a cosmopolitan and polyphagous pest, feeds on more than 300 plant species and responsible for considerable economic damage to many field and horticultural crops (Sachan, 1992; Arora *et al.*, 2005) [11, 2]. The incidence of this insect begins from early vegetative to maturity stage of the crop recording reduction in yield ranging from 40–50% and may cause even total loss of the crop (Rai *et al.*, 2003; Mandal and Roy, 2012) [10, 6]. The annual losses due to insect pests have been estimated up to 15% in chickpea (Chandrashekar *et al.*, 2014) [3]. Crop damage by insect pests could be minimized and kept under economic threshold level effectively by adopting chemical control spray. Such, discriminate use of pesticides lead to problem of development of resistance, resurgence, environmental pollution and health hazards.

Deltamethrin[(S)-cyano-(3-phenoxyphenyl)methyl] (1R,3R)-3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropane-1-carboxylate is a pyrethroid ester insecticide used as an alternative to permethrin, cypermethrin and other organophosphate based insecticides such as malathion and fenthion with exceptional activity against a broad spectrum of lepidopterous insects which act on nervous system. The current investigation was formulated to evaluate the dissipation pattern of deltamethrin to fit in the chickpea pest management strategy.

Materials and Methods**Field Experiment**

Experiment was carried out at student farm, College of Agriculture, PJTSAU, Rajendranagar, Hyderabad, Telangana, India located at 17°19'19.2" latitude, 78°24'39.2" longitude and at an elevation of 534 m above MSL.

The chickpea (var. Jaki 1211), Strong 386 was raised as per the package of practices of PJTSAU, 2015 [9]. The weather parameters recorded during the period of study was given in Table 1.

The chickpea plants were cropped in soil of pH 7.8, EC 0.32, OC 0.42, available nitrogen 210 kg/ha, available phosphorous 44.3 kg/ha and available potassium 351 kg/ha.

Table 1: Meteorological parameters during chickpea crop study

Month & Year	Temperature (°C)		Relative Humidity (%)		Rainfall (mm)
	Min	Max.	I	II	
October, 2015	19.7	30.4	90.6	45.6	11.8
November, 2015	17.6	30.4	88.0	55.3	8.6
December, 2015	14.8	30.9	86.5	34.8	0.0
January, 2016	13.7	29.7	78.9	31.1	0.0
February, 2016	17.4	33.6	78.9	29.6	0.0

A field experiment was conducted during *rabi* season of 2015-16 at PJTSAU, Telangana, India to investigate the dissipation pattern of deltamethrin in immature pods and soil at harvest. The variety selected was Jaki 1211. The trial was laid out in Randomized Block Design with three treatments and replicated thrice in plot size of 6×5 m² each and row and plant spacing of 45×10 cm² and rest of the agronomic

practices were followed as per the Professor Jayashankar Telangana State Agricultural University package of practices. Three foliar sprays were applied starting at pod initiation stage and repeated at 7 days interval with a high volume Knapsack sprayer @ 500 liter spray fluid per hectare. Due care was taken to avoid the drift of insecticides on neighboring plots.

Residue Analysis

Linearity study

Calibration curve was established with concentrations of the standard and corresponding peak area. The regression coefficient (R²) obtained from the curve was greater than 0.99 over the range tested (Fig 1).

The result of the linearity study proved the methods ability to obtain test results, which are directly proportional to the concentration of analyte in the sample.

Concentration	R ₁	R ₂	R ₃	Average
10 ppb	12789	15030	14050	13956
25 ppb	51574	53168	54187	52976
50 ppb	104708	111069	109758	108512
100 ppb	210063	205208	209761	208344
250 ppb	543514	566337	496018	535290
500 ppb	1104406	1065750	1063327	1077828

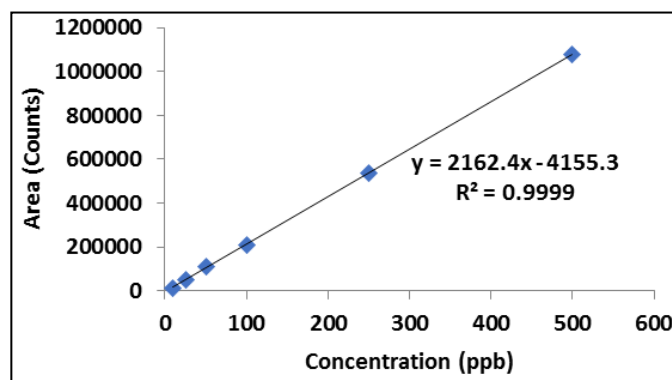


Fig 1: Linearity graph of Deltamethrin

Chemicals and Sampling

Green pods of chickpea were drawn at zero (2 hrs after spray), one, three, five, seven, ten and fifteen days after the final spray.

The samples of 500 g of green pods, mature pods and soil samples from each replication were collected at random and brought to the laboratory for residue analysis. Samples were then ground and prepared for residue determination. The certified reference standard of deltamethrin (purity 99.5%) was supplied by M/s. Bayer Crop Science Limited (Mumbai, India). The suitability of the solvents used in this study was ensured by running them in reagent blanks along with actual analysis. Acetonitrile and water were of HPLC grade. The stock solution of deltamethrin was prepared at the concentration of 1,000 µg mL⁻¹.

Extraction and clean-up for chickpea pods

Homogenized 15±0.1 g of chickpea green pods was taken in 50 ml centrifuge tube and added with 30±0.1 ml acetonitrile and homogenized at 14000 -15000 rpm for 2-3 min using heidolph silent crusher.

The samples were then added with 3±0.1g sodium chloride and mixed by shaking gently followed by centrifugation for 3 min at 2500-3000 rpm to separate the organic layer. The

supernatant organic layer of about 16 ml was taken into 50 ml centrifuge tube and added with 9±0.1 g anhydrous sodium sulphate. 8 ml of extract was taken into 15 ml centrifuge tube containing 0.4±0.01 g PSA sorbent (for dispersive solid phase d-SPE cleanup) and 1.2±0.01 g anhydrous magnesium sulphate.

The sample tube was vortexed for 30 sec then followed by centrifugation for 5 min at 2500-3000 rpm. The extract of about 1 ml Acetonitrile was taken for analysis on LC-MS/MS under standard operational conditions (Table 2).

Extraction and clean-up for soil samples

Homogenized 10±0.1 g soil sample was taken into 50 ml centrifuge tube. The sample tube was then added with 20±0.1 ml acetonitrile, 1±0.1 g sodium chloride and 4±0.1 g magnesium sulphate and mixed by shaking gently followed by centrifugation for 3 min at 3300 rpm to separate the organic layer.

The top organic layer of about 10 ml was taken into 15 ml centrifuge tube containing 1.5±0.1 g magnesium sulphate and 0.25 g PSA and sonicated for 1 min to remove air bubbles and centrifuged for 10 min at 3000 rpm. Extract of about 1 ml Acetonitrile was taken for analysis on LC-MS/MS under standard operational conditions (Table 2).

Table 2: Standard operational parameters of LC-MS/MS

LC-MS/MS	Shimadzu Lcms/Ms - 8040.
Detector	Mass Spectrophotometer
Column	Kinetex, 2.6 μ , C18 Column, 100 x 3.0.
Column oven temperature	40°C
Retention time(min)	6.461
Nebulizing gas	Nitrogen
Nebulizing gas flow	2.0 liters/min
Pump mode/ flow	Gradient / 0.4 ml/ min
Solvents	A: Ammonium Formate In Water(10Mm) B: Ammonium Formate In Acetonitrile (10Mm)
LC programme	Time solvent Conc.
	0.01 B Conc. 60
	2.00 B Conc. 95%
	5.00 B Conc. 85%
	7.00 B Conc. 70%
	9.00 B conc. 60%
10.00 B conc. 60%	
Total Time Programme	10 min
LC-MS/MS Make and Model	SHIMADZU LCMS/MS - 8040.
Software used	Shimadzu Lab Solutions

Rate of Recovery

The average of recovery percentages of deltamethrin from green pods were 110 percent at 0.05 mg/kg; 96.4 percent at 0.25 mg/kg and 89.6 percent at 0.5 mg/kg level whereas in soil it is 111.33 per cent at 0.05 mg/kg; 96.67 percent at 0.25 mg/kg and 90.73 percent at 0.5 mg/kg respectively (Table3).

Table 3: Per cent recovery of deltamethrin in chick pea pods and cropped soil

Substrate	*Percent Recovery (Mean* \pm SD)		
	Fortification Level		
	0.05 mg/kg	0.25 mg/kg	0.50 mg/kg
Chick pea pods	110.00 \pm 3.46	96.40 \pm 2.62	89.6 \pm 1.51
Soil	111.33 \pm 4.16	96.67 \pm 2.81	90.73 \pm 0.80

* mean of three replications

Table 4: Residues and dissipation of deltamethrin on chickpea and soil

Days after treatment	Residues of deltamethrin (mg/kg)										
	X Dose – 12.5 g a.i./ha					X Dose – 25 g a.i./ha					
	R1	R2	R3	Mean \pm SD	Dissipation (%)	R1	R2	R3	Mean \pm SD	Dissipation (%)	
GREEN PODS	0	0.710	0.620	0.674	0.668 \pm 0.045	0.00	1.440	1.380	1.439	1.420 \pm 0.034	0.00
	1	0.502	0.442	0.546	0.497 \pm 0.052	25.60	1.190	1.164	1.152	1.169 \pm 0.019	17.68
	3	0.347	0.354	0.386	0.362 \pm 0.021	45.80	0.745	0.827	0.730	0.767 \pm 0.052	45.99
	5	0.128	0.123	0.137	0.129 \pm 0.007	80.68	0.328	0.269	0.288	0.295 \pm 0.030	79.23
	7	0.057	0.061	0.060	0.059 \pm 0.002	91.20	0.154	0.148	0.147	0.150 \pm 0.004	89.44
	10	<LOQ	<LOQ	<LOQ	<LOQ	-	0.081	0.083	0.082	0.082 \pm 0.001	94.23
15	<LOQ	<LOQ	<LOQ	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	-	
Mature pods (At harvest time)	<LOQ	<LOQ	<LOQ	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	-	
Soil at 30 th day	<LOQ	<LOQ	<LOQ	<LOQ	-	<LOQ	<LOQ	<LOQ	<LOQ	-	
Regression equation	Y=-0.269X+3.200					-	Y=-0.265X+3.539				
R ²	R ² =0.938					-	R ² =0.967				
Half life	1.123					-	1.135				

Kinetic Study

To calculate the rate of degradation, waiting period and half-life of deltamethrin on green pods of chickpea, Hoskin's (1961) [5] linear regression equation was followed. The period to be allowed to expect the residues to reach below the tolerance limit after treatment was calculated by using the formula (Gunther and Blinn, 1955) [4].

Y = a + b X where,

Y - Log of tolerance limit

a - Log of initial deposit

b - Slope of the regression line

Results and Discussion

Dissipation studies

The dissipation of deltamethrin in green pods at an interval of zero (2 hr after spray), one, three, five, seven, 10 and 15 days after last spray were presented in Table 4. An initial deposit of 0.668 mg kg⁻¹ was gradually dissipated to 0.497, 0.362, 0.129 and 0.059 mg kg⁻¹ with the percent dissipation of 25.60, 45.80, 80.68 and 91.20 on one, three, five and seven days, respectively at 12.5 g a.i./ha with half life of 1.123 days. Whereas in case of double the dose (25 g a.i./ha), the initial deposit of 1.420 mg/kg of deltamethrin was detected in green pod samples which dissipated to 1.169 mg/kg by 1st day, 0.767 mg/kg by 3rd day, 0.295 mg/kg by 5th day, 0.150 mg/kg by 7th day, 0.082 mg/kg by 10th and below limit of quantification (<0.05 mg/kg) by 15th day (Table 4 & Fig 2) with half-life of 1.135 days respectively.

The residues of deltamethrin in chickpea mature pods and soil samples collected at harvest from both the doses were found to be below limit of quantification (<0.05 mg/kg)

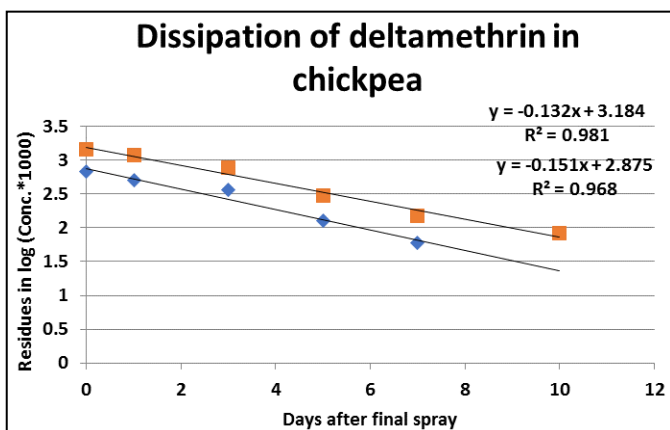


Fig 2: Semi logarithmic graph depicting dissipation dynamics of deltamethrin in Chickpea pods

The present findings are in agreement with research reports to that of Suneet *et al.*, 2012^[14], when deltamethrin 10 EC was applied @17.5 and 35 g *a.i.* ha⁻¹ and samples of green chilli were drawn at different time intervals and that of red chili and soil at harvest time and quantified. Half-life of deltamethrin at application rate of 17.5 g *a.i.* ha⁻¹ varied from 0.36 to 1.99 days and at double the application rate was found to range from 0.38 to 2.06 days. Residues of deltamethrin were found below its determination limit of 0.01 mg kg⁻¹ in red chili and soil at harvest respectively. The results were in accordance with that of Patil *et al.*, 2018^[8] whose results revealed that average initial residues of deltamethrin in immature onion were found to be 1.30 and 2.33 mg/kg at single and double dose, respectively and reached BDL on 10 and 15 days, respectively for both the doses. The half-life values of deltamethrin were observed to be 1.57 and 1.77 days, for single and double doses, respectively. The residues of deltamethrin in mature onion and in soil at harvest were found to be LOQ. Soil sample collected after harvest did not reveal the presence of deltamethrin residues. Minimum persistence was observed in the deltamethrin, only 1 to 3 days in pods and 3 to 7 days in leaves (Srivastava *et al.*, 2002)^[13]. Multi-location supervised field trials to study the persistence, dissipation and risk assessment of combi insecticide flubendiamide and deltamethrin on cucumber (*Cucumis sativus*) was conducted by Sharma *et al.*, 2018^[12]. Residues of flubendiamide and deltamethrin on cucumber resulting from three spray applications of a combination formulation (flubendiamide 90% + deltamethrin 60%, 150 SC) at recommended (22.5 + 15 g *a.i./ha*) and double the recommended (45 + 30 g *a.i./ha*) dose were analyzed. On the basis of persistence and dissipation studies, the half-life ($T_{1/2}$) of flubendiamide on cucumber varied from 1.40 to 2.98 (recommended dose) and 1.55 to 2.76 days (double the recommended dose), while that of deltamethrin ranged from 2.5 to 4.9 (recommended dose) and 2.7 to 3.9 days (double the recommended dose) at the four locations. On the basis of supervised field trial data and using OECD calculator, MRLs in the combination product of 3 mg kg⁻¹ for flubendiamide and 1.5 mg kg⁻¹ for deltamethrin has been proposed for consideration by the Food Safety and Standards Authority of India (FSSAI). Codex, EU and EPA have fixed MRL of 0.2 mg kg⁻¹ for flubendiamide and deltamethrin.

Conclusion

The study clearly showed that deltamethrin when used as foliar spray, did not record residues (<LOQ) in mature pods

and soil 15 days after treatment. Hence, deltamethrin may be recommended as foliar spray to protect chickpea pods from pod borer, without the problem of presence of residues in mature seeds and soil at harvest.

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