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Persistence studies of bifenthrin and lambda cyhalothrin residues in/on Green chilli

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Abstract
Persistence studies of bifenthrin 10% EC and lambda cyhalothrin 5% EC in / on green chilli was undertaken by following two foliar applications at recommended dose (50 g *a.i.*/ha; 15 g *a.i.*/ha) at fruiting stage of the crop. The residues of bifenthrin and lambda cyhalothrin dissipated to below limit of quantification (LOQ) of 0.05 mg/kg on 7th and 10th day with half-life of 2.89 and 5.01 days respectively. Considering this, waiting period of seven to ten days can be suggested for bifenthrin and lambda cyhalothrin with reduced risk of insecticide residues in green chilli.

Keywords: Green chilli, bifenthrin, lambda cyhalothrin, persistence, half life, QuEChERS

Introduction
Chilli (*Capsicum annum* L.), a member of solanaceae family, is one of the most valuable spice crop grown in India and chiefly popular for its green pungent fruits which are used both green and ripe to impart tanginess to vegetable and non-vegetable food items. It is a rich source of minerals like molybdenum, manganese, potassium, copper and vitamin A, B, C and E (Thamaraikannan *et al.*, 2011) [1]. India is the largest producer, consumer and exporter of Chilli, cultivating in the area of 2, 87,000 ha with 3,406 metric tons production (Horticulture at a Glance, 2017) [2]. In Telangana it is cultivated in an area of 0.85 lakh ha with production of 3.28 lakh tons and productivity of 3859 kg/ha (Horticulture Statistics, 2018) [2]. Although there are number of factors responsible for decreasing the yield of chilli but incidence of various insects and non-insect pests are the major threats for declining in the production and productivity of chilli. They comprises of more than 39 genera and 51 species of insects and mite species in the field as well as in the storage (Hosamani *et al.*, 2005) [8]. Aphids (*Aphis gossypii* Glover), thrips (*Scirtothrips dorsalis* Hood) and jassids (*Amrasca bigutula bigutula* Ishida) are some major insect pests of chilli (Jadhav *et al*., 2004 and Chintkuntlawar *et al.*, 2015) [9, 10]. Out of the various insect and non-insect pests attacking chilli, the thrips, *Scirtothrips dorsalis* Hood (Thripidae: Thysanoptera) (Bhede *et al.*, 2008b) [11] and chilli mite *Polyphagotarsonemus latus* (Moghe,1977 in Maharashtra; Das and Roy choudhury, 1978 and Sarkar *et al*., 2008 in gangetic alluvial plains of West Bengal) [12, 13, 14, 15] are considered the most destructive pests in India. Thrips alone is reported to be a major pest of chilli in south India (Dharmasena, 1998) [15], Madhya Pradesh (Patel and Khatri, 1982) [16] and Gujarat (Patel *et al*, 1983) [17]. Among them occurrence of viral diseases as well as ravages caused by insect pests are significant ones (Gundannavar *et al.*, 2007) [18].
Farmers are using insecticides even at picking stage, due to which the residues of noxious insecticides are left in/on fruits, which are very hazardous to consumer health. The constant intake of these left over chemical substances although in minute amount can result in their deposition in the human body, causing ill effects on human health. The persistence of an insecticide varies with the nature of insecticides, dosage applied, numerous applications, crop variety and agro climatic conditions. Before any pesticide is recommended for use in the field it is obligatory to study its residues on the crop so that its efficacy against pest along with its toxicologically acceptable deposit in food products can be acknowledged. There is a need to generate dissipation data for the pesticides which are commonly used by farming community therefore the present study has been contemplated to study the persistence of bifenthrin and lambda cyhalothrin in chilli for southern telangana region which will be useful for inspecting persistence and dissipation behaviour of selected pesticides there by to suggest half life and safe waiting period.

Materials and Methods
Field experiment
Residues and persistence studies of bifenthrin and lambda cyhalothrin in green chilli was undertaken during *kharif* 2016 at the Student Farm, College of Agriculture, PJTSAU,

Rajendranagar, Hyderabad. Chilli seedlings were transplanted to raise the healthy crop by following recommended package of practices. The crop was sprayed with bifenthrin 50 g. *a.i.*/ha and lambda cyhalothrin 10% EC @ 15 g. *a.i.*/ha. Two sprays of each insecticide were given separately at an interval of 10 days starting from fruiting stage.

Chemicals and Reagents

Certified Reference Materials (CRM) of bifenthrin (98.5% purity) and lambda Cyhalothrin (96.9% purity) procured from M/S Sigma Aldrich, Germany were utilized for making primary, intermediary and working standards using GC PR grade acetone and hexane as solvents. Working standards were prepared in the range of 0.01 ppm to 0.5 ppm in 10 mL calibrated graduated volumetric flask using distilled n-hexane as solvent. Primary Secondary Amine (Agilent), magnesium sulfate anhydrous (Emsure grade of Merck), sodium sulfate anhydrous (Emparta ACS grade of Merck), acetonitrile (HPLC gradient grade of Merck), acetic acid glacial (HPLC grade of Merck), acetone (Emplura grade of Merck), n-hexane (HPLC grade of Merck) were used during the study for sample preparation. Bifenthrin 10% EC and lambda cyhalothrin 5% EC formulations were procured from local market.

Residue analysis

Standard preparation

An accurately weighed 10 mg of an individual standard was dissolved in 10 ml volumetric flask using toluene to prepare the standard stock solution of 1000 mg kg^{-1}. Standard stock solution of each insecticide was serially diluted to obtain intermediate lower concentration of 100 mg kg^{-1}. They were stored in Sanyo Biomedical refrigerator at -40ºC. From intermediate standards, working standards were prepared by suitably diluting the stock solution in n-hexane and used as standard check in analysis, linearity and recovery studies.

Sample collection

The green chilli samples (1kg) were collected at random from each replicate of the treated and control plots separately at regular time interval of 0 (2 hrs after spraying), 1, 3, 5, 7, 10 and 15 days after the final spray. The collected samples were brought to the laboratory in polythene bags and processed immediately.

Method validation

Prior to field experiments, QuEChERS (Quick Easy Cheap Effective Rugged Safe) method for extraction and clean up was validated as per SANCO/12571/2013 guidelines. Chilli fruits (5 kg) collected from control plots were homogenized with high volume homogenizer (Robot Coupe Blixer 6 L) and 15 g was taken in to 50 mL centrifuge tubes. The required quantity of bifenthrin and lambda cyhalothrin intermediary standards were added to each 15 g sample to get fortification levels of 0.05, 0.25 and 0.50 mg kg^{-1} in three replications each respectively. 30±0.1 mL acetonitrile was added to the tube, and sample was homogenized for 2-3 min using Heidolph silent crusher (low volume homogeniser). Then 3±0.1g sodium chloride was added to tube and mixed by shaking gently, and centrifuged for 5 min at 2500-3000 rpm with Remi R-238 to separate the organic layer. The top organic layer of about 16 mL was taken into the 50 mL centrifuge tube to which 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 g anhydrous magnesium sulphate, and the sample tube was vortexed for 30 sec followed by centrifugation for 5 min at 3000 rpm. The extract of (2mL) was transferred into test tubes and evaporated to dryness using Turbovap LV of Caliper life sciences with nitrogen gas and reconstituted with 1mL n-Hexane: Acetone (9:1). Chilli samples fortified with lambda cyhalothrin at 0.05, 0.25 mg kg^{-1} and 0.5 mg kg^{-1} were analyzed and the mean recovery of the residues calculated for applying recovery factor while calculating the residues in samples. Fortification and recovery test results are presented in Table 3. The residues detected below 0.05 mg kg^{-1} were mentioned as levels Below Limit of Quantitation (LOQ) in all cases. Per cent recovery was calculated by using following formula.

$$\text{Per cent recovery} = \frac{\text{Quantity of pesticide recovered}}{\text{Quantity of pesticide added}} \times 100$$

Extraction and clean up procedure

2 kg of chilli fruits were collected randomly from each plot in polythene bags. The samples were homogenized with robot coupe blixer. 15±0.1g of homogenized sample was taken in 50 ml centrifuge tube and 30±0.1 ml acetonitrile was added. The sample was homogenized at 14000-15000 rpm for 2-3 min using Heidolph silent crusher and 3±0.1g sodium chloride was then added and mixed by shaking gently followed by centrifugation 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 and added with 9±0.1g anhydrous sodium sulphate 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 g 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 2 ml was transferred into test tubes and evaporated to dryness using Turbovap with nitrogen gas generator and reconstituted with 1 ml n-Hexane for GC analysis with ECD under standard operational conditions.

Table 1: Standard operating parameters for Bifenthrin

Gas Chromatograph	AGILENT GC- 7890B.
Detector	Electron Capture Detector (ECD)
Column	GC capillary column, 35-MS 30mts, 0.25 mm ID, 0.25 µm Film Thickness
Injector temperature	280ºC
Injector status	Split 2
Carrier gas	Nitrogen
Carrier gas flow	2.0 ml/min
Column oven temp	200 ºC-6 min hold-20º /min, 280ºC-@10ºC/min-10 min; TOTAL 20 min
ECD temperature	300ºC
Makeup flow	35 ml/min
Retention time (RT)	12.05 min

Table 2: Standard operating parameters for Lambda Cyhalothrin

Gas Chromatograph	AGILENT GC- 7890B.
Detector	Electron Capture Detector (ECD)
Column	GC capillary column, 35-MS 30mts, 0.25 mm ID, 0.25 µm Film Thickness
Injector temperature	280ºC
Injector status	Split 2
Carrier gas	Nitrogen
Carrier gas flow	2.0 ml/min
Column oven	200 ºC-6 min hold-20º /min, 280ºC-@10ºC/min-10 min; TOTAL 20 min
ECD temperature	300ºC
Makeup flow	35 ml/min
Retention time (RT)	13.12 min

Results and Discussion

The detector response to the neat standards of the insecticides was studied by injecting six linear concentrations of the different insecticides. The graph was plotted with detector response against respective concentrations and linearity line was drawn. The response of the instrument was linear over the range tested and R^2 value was 0.99 for the two insecticides (Fig. 1). These results indicated that the GC-ECD analysis is a valid method for residue determination of the tested insecticides in chilli fruits. Accuracy of the analytical method was determined by recovery studies. The per cent recovery was within acceptable range of 70-120 per cent prescribed by SANCO (2013) and mentioned in Table 3.

Table 3: Recovery of different insecticides in chilli

Fortification level	*Recovery (%)	
	Bifenthrin 5% EC	Lambda cyhalothrin 10% EC
0.05 mg/kg	100.67 (±6.43)	109.33 (±16.29)
0.25 mg/kg	100 (±10.58)	100 (±26.24)
0.5 mg/kg	108.67(±6.43)	111.33 (±9.02)

*Mean of three replications; Figures in parenthesis are SD values

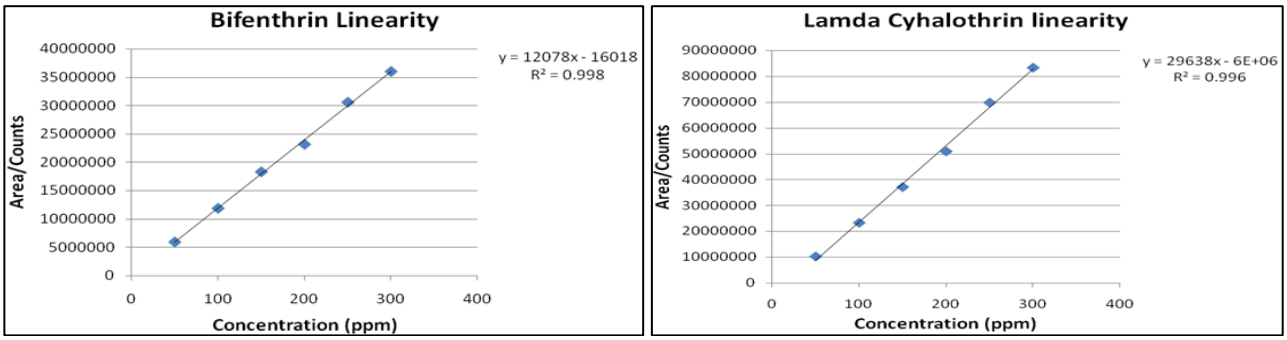

Fig 1: Linearity of bifenthrin and lambda cyhalothrin standard

Dissipation of insecticides

The results revealed that there was reduction in residue levels of the two insecticides in/on chilli with time (Table 4). No residues were recorded in any of the chilli samples collected from untreated plots. At recommended dose of 50 g *a.i.* ha-1, mean initial residues of bifenthrin were 0.433 mg/kg (2 hours after final spray) in green chilli samples which dissipated to 0.332, 0.138, 0.098 and 0.069 mg/kg at 1st, 3rd, 5th and 7th day respectively and reached LOQ at 10 days. Per cent reduction in residues was 23.33, 68.13, 77.37 and 84.06 per cent on above days with half life of 2.89 days. Thomas *et al.*, 2016 reported that mean initial deposit of 0. 56 mg/kg of bifenthrin residues in capsicum dissipated with time and reached below detectable level of 0.05 mg kg^{-1} within 20 days with a half life of 2.18 days at Vellayani, Kerala.

The initial deposits of lambda cyhalothrin in green chilli fruit samples were 0.225 mg/kg which dissipated to 0.168, 0.094, 0.084, 0.063 and 0.054 mg/kg by 1st, 3rd, 5th, 7th and 10th day after final spray and to < LOQ by 15th day whereas per cent reduction in residues was 25.33, 58.22, 62.67 72.00 and 76.00 per cent on above days with half life of 5.01 days. Raghu *et al.*, (2017) documented that in a field trial conducted with lambda cyhalothrin in capsicum both under open field and poly house conditions the residues dissipated to BDL by 5th and 10th day respectively in Telangana state.

Table 4: Dissipation of bifenthrin and lambda cyhalothrin in chilli

Days after final spray	Control	Bifenthrin 5% EC 50 g *a.i.*/ha		Lambda Cyhalothrin 10% EC @ 15 g *a.i.*/ha	
		*Residues (mg/kg) ±SD	Dissipation (%)	*Residues (mg/kg)±SD	Dissipation (%)
0 day	ND	0.433 (±0.048)	-	0.225 (±0.006)	-
1	ND	0.332 (±0.01)	23.33	0.168 (±0.005)	25.33
3	ND	0.138 (±0.001)	68.13	0.094 (±0.006)	58.22
5	ND	0.098 (±0.008)	77.37	0.084 (±0.004)	62.67
7	ND	0.069 (±0.006)	84.06	0.063 (±0.005)	72.00
10	ND	<LOQ	-	0.054 (±0.005)	76.00
15	ND	<LOQ	-	<LOQ	-
Red Chilli (at harvest)	ND	<LOQ	-	<LOQ	-
Regression equation	-	Y = -0.104x + 2.571		Y = -0.060x + 2.265	
R^2	-	0.964		0.902	
Half life (days)	-	2.89 days		5.01 days	

ND – Not Detected; LOQ- Limit of Quantitation (0.05 mg/kg) *Mean of three replications; Figures in parenthesis are SD values

Conclusion

There are no MRLs suggested for bifenthrin and lambda cyhalothrin in chilli either by Codex Alimentarius Commisison or by Food Safety Standards Authority of India. These results clearly suggest that waiting period of 7-10 days must be maintained after spraying of bifenthrin and lambda cyahlothrin on chilli crop considering 0.05 mg/kg as MRL for harvesting green chilli fruits free from residues.

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References

1. Thamaraikannan M, Palaniappan G, Sengottuvel C. Time to step up chilli exports, Market Survey 2011,30-35.
2. Horticulture at a glance. Horticulture Statistics Division Department of Agriculture 2017.
3. Cooperation & Farmer's Welfare, Ministry of Agriculture & Farmers' Welfare, Government of India 490.
4. Raghu B, Shashi Vemuri, Ch. Sreenivasa Rao and S. Swarupa. Disssipation Pattern of Lambda Cyhalothrin on Chilli in Poly house and Open field. Journal of Global Sciences 2017;6(4):4901-4907.
5. Thomas George, Ambily Paul, George Xavier, N Pratheesh Kumar, IV Mithra and SA Sreekutty. 2016. Persistence of Bifenthrin and ƛ-cyhalothrin in Chilli (*Capsicum annuum* L.). Abstract submitted in National Symposium on Agrochemicals Research and Education in India: Appraisal and Road Map for Future.
6. Sanco. Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed 2013. SANCO/ 12571/ 2013 Supersedes SANCO/ 12495/ 2011 Implemented by 01/01/2014.
7. PJTSAU. Vanakalam (*kharif*) 2020-21. Pre-sowing price forecast of chilli 2020.
8. Hosamani AC, Thulasiram K, Patil BV, Bheemana M, Hanchinal SG. Fenpropathrin (Meothrin) 30 EC an ideal insecticide for chilli (*Capsicum annum* L) pest management. Pestology 2005;24(2):21-24.
9. Jadhav VR, Wadnerkar DW, Jayewar NE. Fipronil 5% SC: an effective insecticide against sucking pests of chilli (*Capsicum annum* L). Pestology 2004;28(10):84-87.
10. Chintkuntlawar PS, Pawar UA, Saxsena AK. Insect pest complex of chilli, Capsicum annum L. and their natural enemies in Jabalpur (M.P.). International Journal of Plant Protection 2015;8(2):270-278.
11. Bhede BV, Suryawanshi DS, More DG. Population dynamics and bioeficacy of newer insecticide against chilli thrips, Scirtothrips dorsalis (Hood). Indian Journal of Entomology 2008b;70(3):223-226
12. Moghe PG. Investigations into causes of Churda murda (malformation) disease of chilli in Vidarbha. Current Science 1977;46:631-632.
13. Das LK, Roychoudhury DN. Reaction of jute varieties (Corchorus capsularis) to the yellow mite, Polyphagotarsonemus latus(Acari: Tarsonemidae). Acrological Newsletter 1978;6:2-4.
14. Sarkar H, Mahato S, Somchoudhury AK, Sarkar PK. Management of Yellow mite, Polyphagotarsonemus latus(Banks) infesting chilli (*Capsicum annum* L.) in Gangetic alluvial plains of West Bengal. Journal of Entomological Research 2008;32:127-30.
15. Dharmasena CMD. Present status of managing chilli leaf curl complex in the North Central Province of Sri Lanka. Tropical Agricultural Research and Extension 1998;1(2):154-158.
16. Patel RK, Khatri AK. Note on efficacy of insecticides against chilli thrips. J.N.K.V.V. Research Journal 1982;16(3):274-275.
17. Patel JR, Patel RC, Amin PR. Efficacy of systemic insecticides on thrips and mites and its relation to leafcurl disease of chilli. Gujrat Agricultural University Research Journal 1983;8(2):129-133.
18. Gundannavar KP, Giraddi RS, Kulkarni KA, Awaknavar JS. Development of integrated pest management modules for chilli pests. Karnataka Journal of Agricultural Science 2007;20(4):757-760.