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Research Paper

DISSIPATION PATTERN OF LAMBDA CYHALOTHRIN ON CHILLI IN POLYHOUSE AND OPEN FIELD

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Abstract

Lambda cyhalothrin has a high activity against wide range of chewing and sucking insects, particularly lepidopterons and mites on chilli in India. It is synthetic pyrethroid insecticide and acaricide, most commonly used against both sap sucking and chewing insects and mites due to its contact and acaricidal action. Since chilli is an important component of daily food, food safety issues are very essential As per the ICAR recent suggestions, the studies were conducted to compare the dissipation dynamics of pesticide residues in open field situations and poly house situations so as to suggest PHIs for addressing the food safety issues,when applied twice @15 g a.i. ha-1, first spray at fruit initiation followed by second spray at 10 days interval as per the farmers practice. Lambda cyhalothrin residues were quantified through regular sampling till the residues fell below determination level (BDL) of 0.05 mg kg-1 following the validated QuEChERS method. Initial deposits of 0.37 mg kg-1 detected in chilli samples collected from poly house, dissipated to BDL by 10th day with half-life of 19.8days. In open fields, deposits of 0.16 mg kg-1 dissipated to BDL by 7th day with half-life of 34.65 days, indicated that dissipation is slow in poly house compared to open fields due to various factors,There are no MRLs suggested for lambda cyhalothrin in chilli by either Codex Alimentarius Commission (CAC) or by Food Safety and Standards Authority of India (FSSAI), and hence based on the present study, in poly house and open field PHI of 7 days and5 days can be recommended as the residues degraded to BDL by 10th day and 7th day.

INTRODUCTION

Chilli (*Capsicum annum* L.), also called “red pepper,” is an important cash crop in India and is used throughout the world as a spice, an important ingredient in day to day curries, pickles and chutnies. 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's largest producer, consumer and exporter of chilli with an area 794.12

Thousand ha and production of 1304.38 m t, respectively. Andhra Pradesh ranks first both in area (210.02 thousand ha) and production (685.15Thousand m t) (NHB, 2013). In recent years, due to better cost benefit ratio, farmers are growing chillies in controlled atmosphere conditions, majorly in poly house, besides regular open fields during crop seasons. Chilli suffers major quantitative and qualitative loss in production due to chilli thrips, *Scirtothripsdorsalis* Hood and yellow mite, *Polyphagotarsonemuslatus*(Banks) and fruit borers *Helicoverpaarmigera* (Hubner). The overall reduction in fruit yield of chilli due to thrips and mites damage is up to 34% (Thania *et al*., 2011).These pests not only cause reduction in yield, but also act as vectors for several viral diseases and cause complete failure of crop. 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 is 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 year (Thacker et al., 2005). Many farm gate chilli samples showed presence of insecticide residues (Singh etal.,1999). Lambda cyhalothrin3-(2-chloro-3,3,3-trifluro-1-propenyl)-2,2-dimethyl-cyano(3-phenoxy phenyl)methyl cyclopropanec carboxylatean synthetic pyrethroid insecticide and acaricide, is the most commonly used against both sap sucking and chewing insects and mites due to its contact and acaricidal action. Since chilli is an important component of daily food, food safety issues are very essential.In recent years due to the support of the Government under National Horticulture Mission (NHM), chilli is widely cultivated under poly house conditions. As per Central Insecticides Board and Registration Committee (CIBRC), lambda cyhalothrin is recommended for use on chilli but no MRLs are set. Since the persistence and dissipation of insecticides in poly houses will be different from normal conditions, the present study is proposed to monitor the usage on chilli, dissipation studies of lambda cyhalothrin both in poly house and open field conditions and also evaluation of decontamination methods for removal of lambda cyhalothrin residues, so as to recommend the safe waiting periods based on the Maximum Residue Limits (MRLs) calculated, as it helps in recommending risk mitigation protocols for food safety.

MATERIALS AND METHODS

Chemicals and Reagents: Certified Reference Materials (CRM) of Lambdacyhalothrin (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 of 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 (Emplure grade of Merck), n-hexane (HPLC grade of Merck) were used during the study for sample preparation. Lambda cyhalothrin 50% EC was procured from local market.

Analytical Instruments and Limits of Detection

Working standards were injected in Gas Chromatograph (Agilent 7890 B) with Electron Capture Detector (ECD) and Thermionic Specific Detector (TSD) with injector split ratio of 1:10 using VF-5ms Capillary Column) and confirmatory analysis was done on Bruker Scion 436 GC-MS/MS Triple Quadrupole Detector (EI) using Multiple

Reaction Monitoring (MRM) method (Qualifier ions: 339>188, 339>251, 339>269, 139>97; Quantifier Ions: 139>97). It was found that the limit of detection for lambda cyhalothrin is 0.05 ng in GC-TSD with linearity range of 0.05 ng to 5 ng.

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 7L) and 15 g was taken in to 50 mL centrifuge tubes. The required quantity of lambdacyhalothrin intermediary standards was added to each 15 g sample to get fortification levels of 0.50 mg kg-1 and 0.25 mg kg-1 in three replications each. 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 3 min at 2500-3000 xg 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 2500-3000 xg. The extract of (2mL) was transferred into test tubes and evaporated to dryness using concentration work station (Turbovap LV of Caliper life sciences) with nitrogen gas and reconstituted with 1mL n-Hexane: Acetone (9:1) for dimethoate analysis. 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 1 and the method followed for qualitative and quantitative estimation of lambda cyhalothrin is suitable up to 0.5 mg kg-1 levels as the recoveries obtained are 91.69%,95.88% and 109.66% 0.05,0.25 and 0.50 mg kg-1 fortification level. The residues detected below 0.05 mg kg-1 were mentioned as levels Below Determination Level (BDL) in all cases.

Table 1. Recovery of lambda-cyhalothrin residues inchilli

Details	**Recoveries of lambda-cyhalothrin from fortified chilli samples**					
	Fortified level (mg kg^{-1})					
	0.05 mg kg^{-1}		0.25 mg kg^{-1}		0.50mg kg^{-1}	
	Residues recovered (mg kg^{-1})	Recovery %	Residues recovered (mg kg^{-1})	Recovery %	Residues recovered (mg kg^{-1})	Recovery %
R1	0.044	87.99	0.255	102.06	0.574	114.77
R2	0.046	92.01	0.236	94.40	0.562	112.31
R3	0.048	95.06	0.228	91.18	0.502	100.41
Mean		91.69		95.88		109.16
SD		3.542		5.590		7.678
RSD		3.863		5.830		7.034

Field experiments and sample collections: Chilli crop (Popular hybrid pusajwala) was raised in both poly house and open field in Randomized Block Design at spacing of 60×45 cm with plot size of 20 m^2 and all Good Agricultural Practices (GAPs)

recommended by University were followed. Lambda cyhalothrin5% EC procured from local market was sprayed @15 g a.i. ha-1 twice; first spray at fruit initiation stage followed by second spray at 10 days after first spray, using high volume knapsack sprayer with a spray solution of 500 L ha-1. Pest damage free and crack free chilli fruits of 5 kg were collected from each plot in separate polythene bags and brought to laboratory. Samples were collected at regular intervals i.e. 0, 1, 3, 5, 7, 10, 15, 20 days after last spray for dissipation studies.

RESULTS AND DISCUSSIONS

The dissipation pattern of Lambda cyhalothrin on chilli in open field and poly house situation at an interval of 0, 1, 3, 5, 7, 10, 15 and 20 days after second spray are presented in Table 2 Figs.1 and 2.

The initial deposit of 0.16 mg kg-1 on chilli in open field situations dissipated to 0.12, 0.07 and 0.06 mg kg-1 at 1, 3 and 5 days with per cent dissipation of 25, 56.25 and 62.5, respectively. The half-life (RL50) of Lambda cyhalothrin in chilli fruits was worked out as 34.65 days. The residues of Lambda cyhalothrin dissipated to Below Determination limit of 0.05 mg kg-1 in 7 days. The regression equation was Y = 0.148 + (-0.020) X. There are no MRLs suggested for lambda cyhalothrin in chilli by either Codex Alimentarius Commission (CAC) or by Food Safety and Standards Authority of India (FSSAI), but as per Insecticide Act, 1968, while recommending lambda cyhalothrin @ 15 g a.i. ha^{-1} for fruit borer management, PHI of 5 days was recommended. Since no MRLs are available, PHI of 5 days can be recommend as the residues dissipated to 0.06 mg kg-1.In poly house, Initial deposits of 0.37 mg kg^{-1} lambda cyhalothrin detected at 2 hours after last spray, dissipated to Below Determination Level (BDL) of 0.05 mg kg-1 by 10^{th} day after last spraying on chilli in open field trials. The initial deposits dissipated to 0.12, 0.11, 0.07 and 0.05 mg kg-1 by 1, 3, 5 and 7 days after last spray, respectively. The dissipation pattern showed decrease of residues from first day to 7th day and residues dissipated by 67.56, 70.27, 81.08 and 86.48 % at 1, 3, 5 and 7 days, respectively. The regression equation was Y = 0.256 + (-0.035) X with R^2 of 0.607. There are no MRLs suggested for lambda cyhalothrin in chilli by either Codex Alimentarius Commission (CAC) or by Food Safety and Standards Authority of India (FSSAI), but as per Insecticide Act, 1968, while recommending lambda cyhalothrin @ 15 g a.i. ha^{-1} for fruit borer management, PHI of 7 days was recommended for poly house sprays. Since no MRLs are available, PHI of 7 days can be recommend as the residues dissipated to 0.05 mg kg^{-1}. Since sufficient literature is not available on the dissipation pattern of lambda cyhalothrin in chilli or on related crops in poly houses, discussion part for poly house data is not done. The information available on related crops is also taken into account for discussion of open field data.

The above findings are in agreement with results of Jayakrishnan *et al.* (2005) who reported that lambda cyhalothrin sprayed @ 15 g a.i. ha^{-1} on tomato fruits recorded initial deposit of 0.38 mg kg^{-1} and residues reached Below Determination Level (BDL) by 7^{th} day, with suggested waiting period of 3 days. At double the recommended dose i.e. @ 30 g a.i. ha^{-1}, initial deposit of 0.52 mg kg^{-1} dissipated to BDL by 10^{th} day, with half-life of 3.7 days. The findings of present investigations are also in line with results of Natekar *et al.* (1988) who observed that lambda cyhalothrin sprayed @ 0.005% on brinjal recorded initial deposit of 0.13 mg kg^{-1} and fell Below Determination Level (BDL) by 8^{th} day with a waiting period of 1day.

The dissipation dynamics of lambda cyhalothrin on chilli in both open filed and poly house are in first order kinetics, and are in agreement with the results of Ahuja *et al.*

(2006) on brinjal at both recommended (@ 15 g a.i. ha^{-1}) and double dosage (@ 30 g a.i. ha^{-1}) where the initial deposits of 0.75 mg kg^{-1}, and 1.27 mg kg^{-1} dissipated to BDL in 10 days, respectively. Reddy *et al.* (2007) using lambda cyhalothrin on chilli at 50g a.i. ha-1, recordedinitial deposits of 0.62 mg kg-1. Further, it was also reported that the initial deposits may vary with formulation for the same dosage.

The initial deposits and dissipation vary from crop to crop depending up on the crop canopy, season, age of the crop, sample matrix, surface area of sample etc., and the same can be witnessed based on the test reports published by Pawar *et al.* (1993), Sharma and Awasthi (2002), Singh and Singh (2003) and Ahuja *et al.* (2006) on various crops at different doses.

Table 2. Dissipation of lambda cyhalothrin in chilli

	in poly house						**in open fields**					
Days after last spray	**Residues of lambda cyhalothrin (mg kg^{-1})**					**Dissipation %**	**Residues of lambda cyhalothrin (mg kg^{-1})**					**Dissipation %**
	R1	**R2**	**R3**	**R4**	**Average**		**R1**	**R2**	**R3**	**R4**	**Average**	
0	0.38	0.38	0.35	0.37	0.37	0	0.17	0.16	0.17	0.16	0.16	0
1	0.13	0.12	0.12	0.12	0.12	67.56	0.11	0.11	0.11	0.12	0.12	25
3	0.11	0.11	0.10	0.11	0.11	70.27	0.07	0.07	0.07	0.07	0.07	56.25
5	0.08	0.08	0.06	0.07	0.07	81.08	0.06	0.06	0.05	0.06	0.06	62.5
7	0.05	0.05	0.05	0.05	0.05	86.48	BDL	BDL	BDL	BDL	BDL	100
10	BDL	BDL	BDL	BDL	BDL	100	BDL	BDL	BDL	BDL	BDL	100
15	BDL	BDL	BDL	BDL	BDL	100	BDL	BDL	BDL	BDL	BDL	100
20	BDL	BDL	BDL	BDL	BDL	100	BDL	BDL	BDL	BDL	BDL	100
Regression equation	Y = 0.256 + (-0.035) X						Y = 0.148 + (-0.020) X					
R^2	0.607						0.885					
Half-life	19.8 days						34.65 days					
Safe waiting period : 7 days Codex MRL : NA FSSAI MRL : NA							Safe waiting period : 5days Codex MRL : NA FSSAI MRL :NA					
BDL : Below Determination Level (< 0.25 mg kg^{-1}) NA : Not available							BDL : Below Determination Level (< 0.25 mg kg^{-1}) NA : Not available					

Fig. 1. Dissipation of lambda cyhalothrin in chilli in poly house

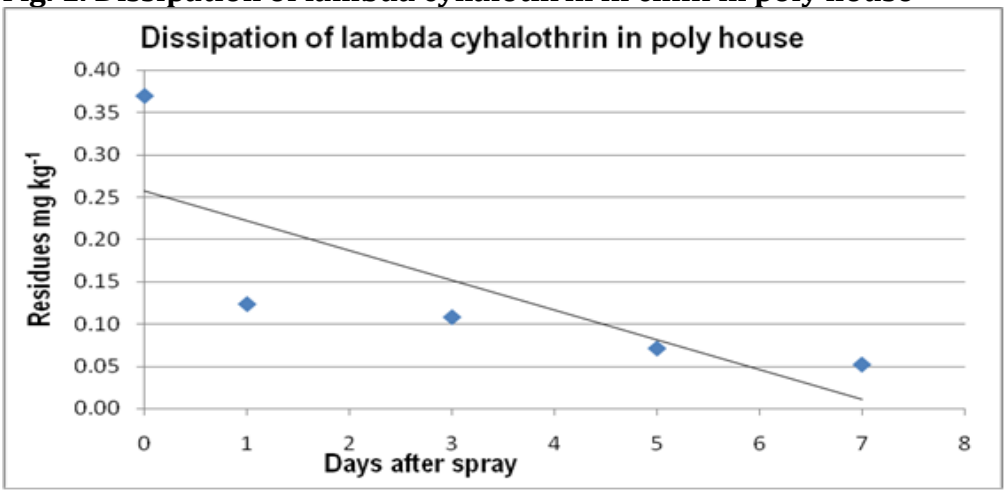
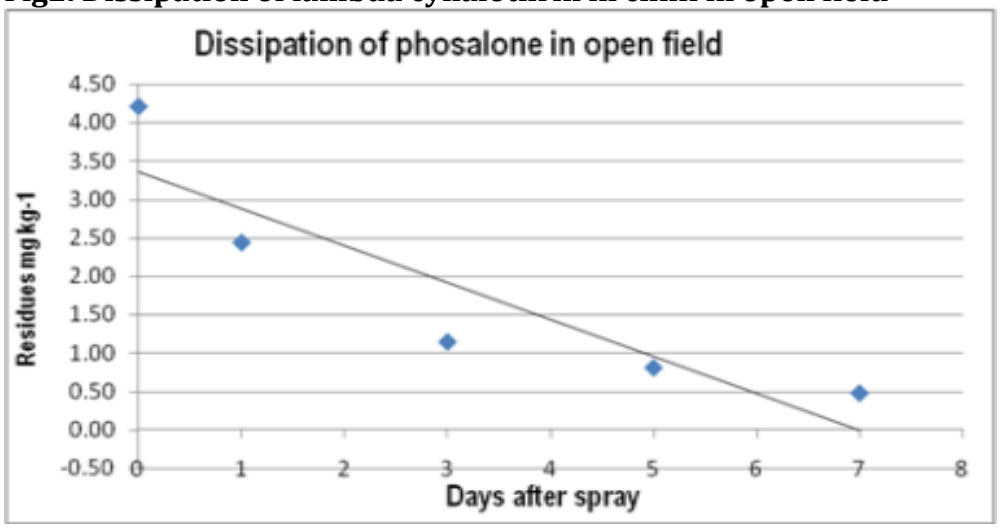


Fig2. Dissipation of lambda cyhalothrin in chilli in open field



CONCLUSION

The dissipation pattern of lambda cyhalothrin varied in poly houses and open fields, where initial deposits were comparatively higher and dissipated slowly in poly houses, Lambda cyhalothrin applied @ 15 g a.i. ha^{-1} recorded initial deposits of 0.37 mg kg^{-1}which dissipated to BDL in 10 days in poly house, while in open field trial, residues dissipated to BDL in 7 days. There are no MRLs suggested by either (CAC) or by (FSSAI), Taking BDL in to consideration PHI of 5 day and 7 day are recommended in open field and poly house respectively.

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