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Research Article

DISSIPATION AND RISK ASSESSMENT OF THIAMETHOXAM 12.6% + LAMBDACYHALOTHRIN 9.5% ZC IN TOMATO

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Abstract: A field experiment was carried out at Kalliyoor panchayat to determine the persistence and dissipation of the insecticide mixture thiamethoxam 12.6% + lambda cyhalothrin 9.5% ZC @ 33+15.75 g a.i ha^{-1} in tomato. The mean initial deposit of thiamethoxam and lambda cyhalothrin were 0.07 and 0.06 mg kg^{-1} and both reached the limit of quantification three days after spraying with half –lives 4.05 and 3.42 days respectively. Risk assessment studies using the dissipation data ensured the safety of the insecticide mixture sprayed product to the consumer. The residue analysis was carried out by LC- MS/MS and GC-ECD.

Keywords: *Dissipation, Risk Assessment, Half-Life, Thiamethoxam, Lambda Cyhalothrin*

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Introduction

Tomato occupies an area of about 78,900 ha with a production of 1,97,59,000 MT [1] in India among the three largest tomato producing countries in the world. The major barrier in the quality production of tomato is the diverse range of pests attacking both the plants and the fruits. In all, 16 pests are recorded to attack tomato from germination to harvest [2]. The population of these pests are subsided by farmers mainly by relying on chemical insecticides that streamed on the plants, ultimately resulting in insecticide resistance [3]. In this contest, insecticide mixtures can play a major role in pest management due to their efficiency in reducing resistance development. Insecticide thiamethoxam 12.6% + lambda cyhalothrin 9.5% ZC belongs to neonicotinoid and synthetic pyrethroid groups respectively with different mode of action for pest management [4] that can control both sucking and chewing pests in tomato. Thus, residue analysis and risk assessment of this insecticide mixture is important, to ensure its safety with reference to consumers.

Materials and Methods

Chemicals and reagents

Certified reference material (CRM) of lambda cyhalothrin, and thiamethoxam were purchased from Sigma- Aldrich Pvt. Ltd. Commercial insecticide mixture of thiamethoxam 12.6% + lambda cyhalothrin 9.5% ZC with trade name Alika was procured from Syngenta Pvt. Ltd. Acetonitrile, water, methanol (HPLC grade), sodium chloride and anhydrous sodium sulphate were supplied from Merck, Germany. Primary secondary amine (PSA) was procured from Agilent technologies, USA. Sodium chloride, anhydrous sodium sulphate and magnesium sulphate were activated in a muffle furnace at a temperature of 350°C for 4 hours and kept in desiccators.

Preparation of standard and validation of analytical method

Standard stock solutions of thiamethoxam was prepared in methanol and lambda cyhalothrin was prepared in n-hexane. Calibration curve was made by injecting the standards prepared from different concentrations (1, 0.5, 0.25, 0.1, 0.05, 0.025, 0.01 µg mL-1) of standard solutions from stock solution by serial dilution. All standard solutions were stored at -20°C before and after use. Recovery studies were conducted before the residue analysis. Untreated tomato fruits were fortified at three different level 0.05, 0.10 and 0.25 mg kg -1. Percentage recovery and relative standard deviation were analysed for accuracy and precision.

Field experiment

One-month old seedling of tomato variety Vellayani vijay was planted and raised in a field at Kalliyoor adhering to the package of practices of Kerala Agricultural University [5]. The experiment was conducted in randomised block design with four treatments and five replications. The insecticide mixture was sprayed two times for residue analysis, first at the time of fruit initiation and second ten day after the first spray. The fruit samples were collected 0, 1, 3, 5, 7, 10, 15 and 30 days after spraying.

Residue analysis

Fruit samples collected were taken to the Pesticide Residues Research and Analytical laboratory, College of Agriculture, Vellayani for residue analysis following the QuEChERS (Quick, easy, cheap, effective, rugged, safe) method [6]. 25g copped, crushed and ground tomato fruit samples were taken in a 250mL centrifuge bottle. The analyte was extracted by adding 50mL acetonitrile of HPLC grade. After proper shaking 10g activated sodium chloride was added. This centrifuge bottle was closed tightly and centrifuged at 8000 rpm for 8 minutes. 16 mL supernatant was pipetted out and transferred to 50 mL centrifuge tube containing 6g activated sodium sulphate. It was then vortexed and 12 mL was pipetted out and transferred to 15 mL centrifuge tube containing 0.2g PSA and 1.2g magnesium sulphate. After mixing it with the help of vortex the mixture was centrifuged at 2500 rpm for 5 minutes. From this mixture 3ml was pipetted for LC-MS/MS analysis and 4 mL for GC analysis into turbo tubes. These tubes were then placed in turbovap which uses a gentle steam of nitrogen at 40°C and 7.5 psi flow for evaporating them. The residue was reconstituted in 1.5mL of methanol and filtered through 0.2 micron PVDF filter prior to estimation in LC-MS/MS and for GC analysis the residue was reconstituted to 1 mL in n-hexane. Residue analysis of thiamethoxam was done with LC-MS/MS and lambda cyhalothrin with GC-ECD.

Persistence of insecticide is generally expressed in terms of half- life. Half- life refers to the time taken to reach half the initial concentration of the insecticides and it was calculated using Hoskins formula [7]

$$T_{1/2} = \log 2 / K_1$$

K1= Slope of regression line

Risk assessment

Residue of insecticide obtained at each day was used for calculating the risk confronted by the buyers while consuming the insecticide treated products. Dietary risk of thiamethoxam and lambda cyhalothrin were estimated using acceptable daily intake (ADI), maximum permissible intake (MPI) and theoretical maximum residue concentration (TMRC). Based on the residue values, TMRC were calculated by multiplying maximum residue level obtained at recommended dose and total intake of food per day (Daily consumption of tomato was considered as 50 g d-1 [8]).MPI is obtained by multiplying acceptable daily intake and average body weight of an Indian adult which was observed to be 60 Kg [9]. If TMRC is less than MPI, the insecticide will not cause any harm to health. The ADI values of thiamethoxan and lambda cyhalothrin are fixed as 0.08 and 0.02 mg kg-1 bw d-1 respectively [10].

Result and discussion

Validation of analytical method

Results for the validation of both the insecticides showed good recovery of 70-120 percent for three different fortified levels and relative standard deviation less than 20 percent depicting reliability on the residue analysis method. The percentage recovery of thiamethoxam was 87.93, 117.67 and 117.46 with relative standard deviation 4.94, 1.06 and 1.53 percentage at three levels of fortification. In lambda cyhalothrin, the mean percent recovery was 100.00, 114.46 and 108.09 at respective fortification levels of 0.05, 0.10 and 0.25µg mL-1 with relative standard deviation of 2.30, 1.42 and 3.55 percent respectively [Table-1, 2].

Table-1 *Percent recovery of lambda cyhalothrin fortified at different levels using modified QuEChERS method*

LOQ (mg kg^{-1})	Recovery (%)			Mean Recovery	RSD
	R_1	R_2	R_3	(%) ± SD	(%)
0.05	100	104	96	100± 2.30	2.3
0.1	116.7	113.8	112.9	114.46±1.62	1.42
0.25	103.6	113	107.68	108.09±3.84	3.55

Table-2 Percent recovery of thiamethoxam fortified at different levels using modified QuEChERS method

LOQ (mg kg^{-1})	Recovery (%)			Mean Recovery	RSD
	R_1	R_2	R_3	(%) ± SD	(%)
0.05	91.4	90.6	81.8	87.93±4.35	4.94
0.1	118	119	116	117.67±1.25	1.06
0.25	120	116.4	116	117.46±1.79	1.53

Table-3 *Persistence and dissipation of insecticides in Tomato*

Days after spraying (DAS)	Thiamethoxam 12.6% +Lambda cyhalothrin 9.5% ZC			
	Thiamethoxam 12.6%		Lambda cyhalothrin 9.5%	
	Mean residue (mg kg^{-1})	Dissipation (%)	Mean residue (mg kg^{-1})	Dissipation (%)
0 (2 h after spraying)	0.07	-	0.06	-
1	0.06	14.28	0.05	16.66
3	LOQ	Dissipation (%)	LOQ	
Half- life (Days)	4.05		3.42	

Table-4 *Risk assessment of thiamethoxam 12.6% + lambda cyhalothrin 9.5% ZC in Tomato*

ADI (mg kg^{-1} bw d^{-1})		Average body weight (kg^{-1})	DAS	Daily consumption (gd^{-1})	MPI (µg Person^{-1} d^{-1})*		Mean residue (mg kg^{-1})		TMRC (µg Person^{-1} d^{-1})	
Thiamethoxam 12.6%	Lambda cyhalotrin 9.5%				Thiamethoxam 12.6%	Lambda cyhalotrin 9.5%	Thiamethoxam 12.6%	Lambda cyhalotrin 9.5%	Thiamethoxam 12.6%	Lambda cyhalotrin 9.5%
0.08	0.02	60	0	50	4800	1200	0.07	0.06	3.5	3
0.08	0.02	60	1	50	4800	1200	0.06	0.05	3	2.5

Residue analysis

The mean initial deposit of thiamethoxam after two hours of spraying was found to be 0.07 mg kg-1 which dissipated to 0.06 mg kg-1 on first day after spraying with dissipation percentage 14.28. On third day the residue reached below the limit of quantification with half- life 3.42 days [Table-3]. The mean initial deposit of lambda cyhalothrin was found to be 0.06 mg kg-1 after two hours of spraying which dissipated to 0.05 mg kg-1 with dissipation percentage of 16.66 on one day after spraying. On the third day, residues got dissipated below limit of quantification which was 0.05 mg kg-1 and half -life calculated was 4.05 days [Table-3]. In a similar study of dissipation of above insecticide mixture in rice, 83-88% of thiamethoxam dissipated within five days with a half-life 5.2 days and with five days residue reached below the limit of quantification in lambda cyhalothrin with half-life 4.81days [11]. More or less similar results was obtained by Hampaiah [12] in cowpea in Kerala. He reported that the initial residue of thiamethoxam and lambda cyhalothrin after two hours of spraying was 0.42 mg kg-1 and 0.19 mg kg-1 respectively which dissipated to 0.08 and 0.06 mg kg-1 respectively in the first day after spraying reached below the limit of quantification by the third day. The half -lives observed were 0.37 days for thiamethoxam and 0.31 days for lambda cyhalothrin.

Risk assessment

In case of thiamethoxam + lambda cyhalothrin, MPI values calculated were 4800 and 1200 µg person-1 d-1 for thiamethoxam and lambda cyhalothrin respectively [Table-4]. TMRC values for lambda cyhalothrin were 3.00 and 2.50 µg Person-1 d-1 for 0th and first day respectively and for thiamethoxam it was 3.50 and 3.00 µg Person-1 d-1 respectively and these values were lower than the MPI values of lambda cyhalothrin and thiamethoxam. Hampaiah [12] has reported similar results agreeing with the safety of the insecticide mixture with lower TMRC value than MPI values.

Conclusion

The low mean residue concentrations of both thiamethoxam and lambda cyhalothrin in tomato fruit after spraying has ensured its acceptance in managing pest complex attacking tomato. Thus, alternating the conventional insecticides with the insecticide mixture having two different mode of actions can aid in managing insecticide resistance. Risk assessment studies of the insecticide mixture thiamethoxam 12.6% + lambda cyhalothrin 9.5% ZC on tomato has also confirmed its safety towards the consumers.

Application of research: To study the dissipation and risk assessment of insecticide mixture to ensure its safety to the consumers

Research Category: Pesticide Residue Analysis

Abbreviations:

LC-MS/MS- liquid chromatography mass spectrometer
GC-ECD- Gas chromatography- electron capture detector
DAS- days after spraying
LOQ- limit of quantification
RSD- Relative standard deviation

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Study area/ Sample collection: Kalliyoor panchayath of Thiruvananthapuram district

Cultivar/Variety name: Tomato-Vellayani Vijay

Conflict of Interest: None declared

Ethical approval: This article does not contain any studies with human participants or animals performed by any of the authors.
Ethical Committee Approval Number: Nil

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