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Dissipation and decontamination of imidacloprid and lambda - cyhalothrin residues in brinjal

■ S.P. GUPTA*, S.P. SINGH, PAIDI SATYANARAYANA AND NAGENDRA KUMAR
Department of Entomology, Rajendra Agricultural University, Pusa, SAMASTIPUR (BIHAR) INDIA

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***Corresponding author:**
Email: surya22gupta@gmail.com

ABSTRACT

Residues ofimidacloprid at 20 g a.i. ha^{-1} and lambda - cyhalothrin at 15 g a.i. ha^{-1} were estimated quantitatively by HPLC/GLC in/on brinjal fruits. Initial deposits of insecticides were higher in imidacloprid than lambda - cyhalothrin. Initial deposits of 0.652 mg kg^{-1} of imidacloprid dissipated to 93.17 per cent on 10th day. In lambda - cyhalothrin the initial deposits were 0.138 mg kg^{-1} which dissipated to 92.75 per cent on 10th day. The degradation of imidacloprid was relatively higher as compared to lambda - cyhalothrin. Both imidacloprid and lambda - cyhalothrin had half - life values of 1.92 and 2.65 days, respectively on brinjal fruits. The safe waiting period for imidacloprid was found 4.70 days. In lambda - cyhalothrin, no waiting period is required after its application as the initial deposits were less than its MRL (0.3 mg kg^{-1}).

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INTRODUCTION

Brinjal (*Solanum melongena*), a native of Indo-Burma region, is one of the important vegetable crops grown as commercial and kitchen garden in India. Brinjal is often described as a poor man's vegetable because it is popular amongst small scale farmers and low income consumers. Not less than 140 species of insect pests have been reported to cause damage to brinjal in India (Frempong and Buhain, 1978). Among all these insect pests, the most serious and destructive one is the shoot and fruit borer, *Leucinodes orbonalis* Guen. (Ghosh *et al.*, 2003). The indiscriminate use of insecticides is causing not only health hazards and ecological imbalance but also polluting the different components of the environment affecting behaviour and habits of organisms. Injudicious use of insecticides also leads to accumulation of toxic residues in food and feed, resistance of insects to insecticides and resurgence of pests etc. A small sized fruit receives comparatively more deposits of toxic insecticides than the medium sized or normal fruit because of its increased surface area per unit weight (Sarode and Lal, 1980; Kashyap and Walia, 1983). However, the application of these insecticides left toxic residues in the final produce and in some case the concentrations were above maximum residues in the final produce and in some case the concentration were above maximum residues limit (Dikshit *et al.*, 1989 and Patel *et al.*, 2001). Therefore, there is a need to replace these insecticides with newer chemicals with lesser dose of few grams per hectare maintaining high toxicity to insect pests.Imidacloprid and lambda - cyhalothrin are highly effective against different pests and the persistence and dissipation of these insecticides were undertaken for the present studies.

MATERIAL AND METHODS

The experiment was laid out in a Randomized Block Design with 7 treatments and 3 replications at the University farm of Rajendra Agricultural University, Pusa during Rabi 2012 - 13. Brinjal cv. RAJENDRA BAIGAN-2 was grown according to the recommended package of practices for this region.The insecticides imidacloprid @ 20 g a.i. ha^{-1} and lambda - cyhalothrin @ 15 g a.i. ha^{-1} were sprayed. The first application of insecticides was done just after the appearance of insect pests and the second and third spray was given at 15 days interval.Brinjal fruit samples of marketable size were collected from each plot a 0 (1 hr after spray), 2, 5, 7, 10 and 15 days after the third spraying. About 1.0 kg sample of brinjal fruits was collected in polyethylene bags from each treatment and replication without touching the fruit by hand. The samples were brought to laboratory for further processing.

Extraction, clean up and estimation :

Imidacloprid :

The samples collected from the field were mixed thoroughly. A representative sample of 50 g was extracted with 100 ml of acetone in a Waring blender for 2 minutes. The pulp was filtered under suction on a Buchner funnel. The process of blending was repeated twice with 75 ml of the solvent and the filtrates were combined. The filtered acetone extract was diluted with distilled water and partitioned with hexane (100 + 50 ml) and then with 50 ml hexane - ethyl acetate (98:2, v/v). The organic phase was discarded to remove the co-extractive. Subsequently, the aqueous phase was extracted three times with dichloromethane (100 + 2 x 50 ml).The dichloromethane phase was washed with aqueous potassium carbonate solution (0.01M , 50 ml) to remove the acidic co-extractives and then dichloromethane phase was dried by passing through 2 cm layer of anhydrous sodium sulphate. The sodium sulphate layer was again washed with 30 ml dichloromethane. The dichloromethane phase containing insecticides residue of imidacloprid as obtained above was evaporated under reduced pressure. The residues were dissolved in 2 ml of ethyl acetate and cleaned up by passing through a glass column packed with 10 g of silica gel over 2 cm layer of anhydrous sodium sulphate. The column was prewashed with 30 ml of ethyl acetate before loading the extract. The column was eluted with 10 ml of ethyl acetate and the elute was discarded. The imidacloprid residue was eluted with 50 ml of acetonitrile and the elute was concentrated under reduced pressure and taken for analysis. The residues were analysed on HPLC (Waters USA) equipped with a UV - VIS (lambda max 270) detector, glasscoloumn Stainless Steel, C-18 and mobile phase Acetonitrile - water (60 : 40). The retention time for imidacloprid was 2.66 minutes.

Lambda - cyhalothrin :

The samples collected from the field were mixed thoroughly. A representative sample of 50 g was extracted with 100 ml of acetone in a Waring blender for 2 minutes. The pulp was filtered under suction on a Buchner funnel. The process of blending was repeated twice with 75 ml of the solvent and the filtrates were combined. The filtered acetone extract was concentrated under reduced pressure and transferred to separatory funnel (500 ml) and diluted with an aqueous solution of sodium chloride (150 ml). The insecticide was partitioned into hexane (50, 25, 25 ml). The hexane layer was passed through anhydrous sodium sulphate and concentrated to a known volume.The hexane extract of insecticide residues representing 25 g of plant material was cleaned up by passing through a glass column packed with florisil and neutral alumina (5 g each) sandwiched between sodium sulphate (2g) layers. The insecticide was eluted with 100 ml mixture of hexane - acetone (9:1, v/v) at rate of 2 - 3 ml min^{-1}. The cleaned up extract was concentrated to dryness and the insecticide residue was dissolved in a known volume of hexane for GLC estimation.The insecticide residues were analysedon chemito GC1000 equiped with aECD - Ni 63 capture detector and Glass column (2m) packed with 3 per cent OV-101 on 80-100 mesh CHW (HP).The column, injector and detector temperatures were 265^0C, 275^0C and 300^0C, respectively. The flow rate of nitrogen carrier gas was 30 ml min^{-1}.

RESULTS AND DISCUSSION

Quantitative estimation of the residues of imidacloprid and lambda - cyhalothrin in/on brinjal fruits was done by high performance liquid chromatographic and gas liquid chromatographic methods, respectively. The mean recovery of imidacloprid and lambda - cyhalothrin in brinjal fruits were found 90.6 and 86.5 per cent, respectively (Table 1).The mean initial deposit of imidacloprid (20 g a.i. ha^{-1}) on brinjal fruits were 0.652 mg kg^{-1} which degraded to 0.420 and 0.214 mg kg^{-1} showing dissipation of 35.58 and 67.18 per cent on 2nd and 5th day of third application, respectively (Table 2). The residues further degraded to 0.116 and 0.040 mg kg^{-1} on 7th and 10th day with a corresponding loss of 82.21 and 93.17 per cent. On 15th day, the residues dissipated to non - detectable level (< 0.006 mg kg^{-1}). The mean initial deposits of lambda - cyhalothrin (15 g a.i. ha^{-1}) on brinjal fruits were 0.138 mg kg^{-1} after application @ 15 g a.i. ha^{-1} which dissipated to 0.085 mg kg^{-1} on 2nd day with a loss of 38.41 per cent. The initial deposits of imidacloprid on brinjal fruits found in the present study are in agreement with the findings of Suganthy *et al.* (2010) who obtained initial deposits of 0.714 - 0.824 mg kg^{-1} and 1.213 - 1.361 mg kg^{-1} on chilli fruits when imidacloprid was applied @ 15 and 25 g a.i. ha^{-1}. The present findings of dissipation of imidacloprid on brinjal fruits are also in close conformation to the reports of earlier workers (Suganthy *et al.*, 2010).

The residues of imidacloprid at 20 g a.i.ha^{-1} and lambda - cyhalothrin at 15 g a.i.ha^{-1} persisted up to 10 days. However, residues of both insecticides became non - detectable on 15th day. The rate of dissipation of

Table 1 : Recovery of imidacloprid and lambda – cyhalothrin in fortified samples of brinjal fruits

Insecticide	Fortification level (mg kg^{-1})	Amount recovered (mg kg^{-1})	Per cent recovery	Mean ± SD
Imidacloprid	1.0	0.891	89.1	
	1.0	0.922	92.2	
	0.5	0.452	90.4	90.6 ± 1.309
	0.5	0.455	91.0	
Lambda - cyhalothrin	1.0	0.842	84.2	
	1.0	0.870	87.0	
	0.5	0.440	88.0	86.5 ± 1.620
	0.5	0.434	86.8	

Table 2 : Residues of imidacloprid in/on brinjal fruits

Dose (g a.i. ha^{-1})	Days after treatment	Residues (mg kg^{-1})				Per cent dissipation
		R_1	R_2	R_3	Mean ± SD	
20	0	0.597	0.620	0.739	0.652 ± 0.076	
	2	0.382	0.468	0.410	0.420 ± 0.044	35.58
	5	0.208	0.240	0.195	0.214 ± 0.023	67.18
	7	0.098	0.131	0.120	0.116 ± 0.017	82.21
	10	0.035	0.056	0.029	0.040 ± 0.014	93.87
	15	ND	ND	ND	ND	

ND = Not Detected

Table 3 : Residues of lambda - cyhalothrin in / on brinjal fruits

Dose (g a.i. ha^{-1})	Days after treatment	Residues (mg kg^{-1})				Per cent dissipation
		R_1	R_2	R_3	Mean ± SD	
15	0	0.125	0.158	0.132	0.138 ± 0.017	-
	2	0.087	0.096	0.073	0.085 ± 0.012	38.41
	5	0.068	0.052	0.049	0.056 ± 0.010	59.43
	7	0.017	0.029	0.020	0.022 ± 0.006	84.06
	10	0.010	0.012	0.009	0.010 ± 0.002	92.75
	15	ND	ND	ND	ND	-

ND = Not Detected

these insecticides was rapid up to 5 days accompanied by gradual to slow at subsequent intervals. Among both insecticides, imidacloprid recorded relatively higher rate of dissipation.Both imidacloprid and lambda - cyhalothrin hadhalf - life values of 1.92 and 2.65 days, respectively. The safe waiting period of imidacloprid on brinjal fruits was found 4.70 days. However, the initial deposits of lambda - cyhalothrin on brinjal fruits were below the MRL (0.3 mg kg^{-1}) and thus no waiting period is required after its application.

The residues of lambda - cyhalothrin on brinjal fruits found in the present study are in agreement with the findings of Singh and Singh (2003) reported initial deposits of 0.335 mg kg^{-1} on chickpea green pods due to lambda - cyhalothrin treatment at 25 g a.i.ha^{-1}. Kumari *et al.* (2005) reported initial deposits of 0.120 and 0.164 mg kg^{-1} on okra fruits following application of lambda - cyhalothrin @ 15 and 30 g a.i. ha^{-1}, respectively.The persistence and dissipation behaviour of the insecticide residues in an agro- climatic condition depend upon a variety of factors such as chemical composition, formulation, dose, plant growth, position of plant part, edaphic, climatic and other related factors (Ebeling, 1963). The present findings showed that the residues of imidacloprid at 20 g a.i.ha^{-1} and lambda - cyhalothrin at 15 g a.i.ha^{-1} persisted up to 10 days. However, residues of both insecticides became non - detectable on 15^{th} day. The rate of dissipation of these insecticides was rapid up to 5 days accompanied by gradual to slow at subsequent intervals. Sharp reduction in residues in first 5 days could be attributed to physical removal by weathering agencies. Gunther and Blinn (1955) reported that initial residues lost due to weathering agencies because of the poor contact with plant material. When log of residues (mg kg^{-1}) was plotted against number of days, it resulted in a straight line, thus indicating that the rate of dissipation of both the insecticides followed a first order reaction.The recovery of the insecticides obtained in the present investigation are also in close agreement to the recovery reports of earlier workers on vegetables and other crops (Gajbhiye *et al.*, 2004; Dharumarajan and Dikshit, 2010 and Kumar *et al.*, 1998).

The data presented in Table 1 showed that both imidacloprid and lambda - cyhalothrin had half - life values of 1.92 and 2.65 days, respectively. The half - life values of imidacloprid obtained in the present study are in agreement to the reports of Suganthy *et al.* (2010) obtained half - life value of imidacloprid (15 and 25 g a.i. ha^{-1}) on chillies as 2.85 - 3.75 days. Half - life values for lambda - cyhalothrin on brinjal fruits found in the present investigation are similar to the findings of Gopal and Mukherjee (1997) who observed a half- life of 4 days on pea pods.

Waiting period, also termed as safety interval, is the number of days to be elapsed before insecticide residues reach its maximum residue limit (MRL). It was calculated for both the insecticides on brinjal fruits on the basis of formula given by Hoskins (1961). The results are presented in Table 1. Imidacloprid and lambda - cyhalothrin have been assigned the MRL values of 0.2 mg kg^{-1} and 0.3 mg kg^{-1}, respectively onbrinjal fruits (Codex Alimentarius Commission, 2012).

Imidacloprid residues were found to dissipate below MRL in 4.70 days on brinjal fruits. However, the initial deposits of lambda - cyhalothrin on brinjal fruits were below the MRL (0.3 mg kg^{-1}) and thus no waiting period is required after its application (Table 3). The waiting period of insecticides obtained in the present study are in close conformity to the reports of earlier workers. Devee and Baruah (2010) has suggested a waiting period of 4 days on rapeseed leaves after imidacloprid application @ 20 g a.i. ha^{-1}. Suganthy *et al.* (2010) found a safe waiting period of 1.24 - 4.56 days for imidacloprid on chillies fruits. Lal (2005) suggested a safe waiting period of 4 -5 days for lambda - cyhalothrin on okra fruits after considering MRL value of 0.05 mg kg^{-1} on okra fruits.

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