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Persistence of quinalphos and ethion in/on brinjal

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Abstract

Studies on the dissipation pattern of quinalphos and ethion in/on brinjal fruits and soil were undertaken by following two foliar applications at recommended and double the recommended doses quinalphos and ethion (250 and 500 g a.i./ha) at fruiting stage. Residues of quinalphos and ethion dissipated with half-life of 2.05, 2.18 and 2.12, 3.21 days, at both the doses respectively. The residues reached below quantification limit (BDL) on 7th day in quinalphos and ethion at the recommended dose. Considering this, Pre-harvest interval (PHI) of seven days can be suggested for quinalphos and ethion with reduced risk of insecticide residues in brinjal.

Keywords: Quinalphos, ethion, persistence and QuEChERS

Introduction

In India, brinjal is being cultivated round the year on an area of about 5.6 lakh ha with a production of 9.6 million tonnes fruits. However, in Maharashtra it is cultivated area of 3500 ha with a production of 4.9 lakh tonnes and productivity of 14 MT per ha. (Anon., 2013) [1].

Brinjal crop was infested by 26 pests in India, whereas 140 pest species belonging to 50 families from 10 orders are reported on this plant all over the world (Vevai, 1970) [9]. In Maharashtra, number of insect pests have been reported to feed on brinjal at various stages of its growth; which is a serious limiting factor in brinjal production. Among these shoot and fruit borer (*Leucinodes orbonalis* Guenee), whitefly (*Bemisia tabaci* Geladius), aphid (*Aphis gossypii* Glover), jassid (*Amrasca biguttula biguttula* Ishida) and thrips (*Scirtothrips solanifoli* Shamsheer) are predominant. However, *Leucinodes orbonalis* Guenee (Pyralidae: Lepidoptera) considered to be major pest and damages the crop throughout the year.

Commercial consideration of this crop unfortunately compelled the growers to use a large amount of pesticides during the entire period of growth even at fruiting stage and sometimes farmers also ignored the recommended waiting period between the harvest and last spray. In some cases the residues of insecticide exceeded its tolerances. The degradation or dissipation of insecticide is influenced by climatic condition, type of application, plant species, dosage, interval between last application and time of harvest (Khay, *et al.*, 2008) [2]. Therefore, it is necessary to study the safe waiting period for consumption of brinjal fruits and persistence of introduced pesticides in crop.

Material and methods**Field experiment**

The experiment was laid out at the Instructional Farm of Post Graduate Institute, M.P.K.V, Rahuri during *Kharif-2015*. Brinjal seedlings were grown on raised beds by sowing disease free seeds of variety 'Phule Arjun'. Seedlings were ready for transplanting on 30th days after sowing. Seedlings transplanted having spacing 90 x 75 cm. Overall two foliar sprays of each insecticide was given at an interval of 10 days starting at fruit initiation stage, two doses of quinalphos (recommended dose 250 g a.i.ha⁻¹ and double the recommended dose 500 g a.i.ha⁻¹) and two doses of ethion (recommended dose 500 g a.i.ha⁻¹ and double the recommended dose 1000 g a.i.ha⁻¹) were evaluated for residues.

Chemicals and reagents

The certified reference material of quinalphos and ethion with purity of 99.7 % and 97.2% were obtained from Sigma Aldrich and commercial formulations (Ekalux 25 EC and Fosmite 50 EC) were purchased from local market of rahuri. HPLC grade ethyl acetate was obtained from M/s. Avantor performance Material India Limited, Thane. PSA and sodium sulphate anhydrous were procured from Agilend Technology, Bangalore and SDFCL, Mumbai, respectively. Working standards were prepared by dissolving reference standards in ethyl acetate.

Residues analysis

Standard preparation

An accurately weighed 10 mg of an individual analytical grade insecticide was dissolved in 10 ml volumetric flask using suitable solvent to prepare the standard stock solution of 1000 mg kg⁻¹. Standard stock solution of each insecticide was further diluted to obtain intermediate lower concentration of 100 and 10 mg kg⁻¹. They were stored in a refrigerator at -40°C. From intermediate standards, were prepared by suitably diluting the stock solution in n-hexane for quinalphos and ethion and used as standard check in analysis, linearity and recovery studies.

Method validation

Prior to analysis of samples, linearity of quinalphos, ethion was established on GCMS. Accuracy and precision of the method was determined by per cent mean recovery and percent relative standard deviation (RSD). The limit of detection (LOD) of quinalphos and ethion was determined by considering a signal-to-noise ratio of three with reference to the background noise obtained for the blank sample. The limits of quantification (LOQ) determined as 3 times of LOD. Brinjal fruits sample (10 g) was taken in 50 ml centrifuge tubes in three replicates each was spiked with quinalphos and ethion separately at the required fortification levels *i.e.* LOQ, 5 x LOQ and 10 x LOQ, adding an appropriate volume of working standard of 10 mg kg⁻¹. The extraction and clean-up was performed using methodology as described under. The per cent recovery was calculated by using following formula.

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

Residue determination

Residue of quinalphos and ethion was performed using GC-MS. Identification of insecticide residue was accomplished by retention time and compared with known standard at the same condition. The quantities were calculated on peak area basis by using following formula.

Residues (mg kg ⁻¹)	Area of sample	X	µl of sample injected	X	conc. of standard (ppm)	X	Final Vol (ml)
	Area of standard		µl of standard injected		wt. of sample (g)		

$$\text{Wt. of sample (g)} = \frac{\text{Sample Wt. (g)} \times \text{Aliquot taken (ml)}}{\text{Volume of solvent added (ml)}} = \text{gm}$$

Statistical Analysis

The simple statistical analysis was carried out in the Microsoft Excel programmed with the help of computer. The mean residues, standard deviation, regression equation, R² value and half life values were calculated in excel programme.

Sample Collection

The medium marketable size brinjal fruit samples (1 kg) were collected from each plots and control plots separately at regular time interval of 0 (2 hrs after spraying), 1, 3, 5, 7, 10 and 15 days after the second spray. The collected samples (brinjal fruits) were transferred immediately to the laboratory in an ice box. The collected brinjal samples were brought to the laboratory in polythene bags and processed immediately.

Extraction and cleanup

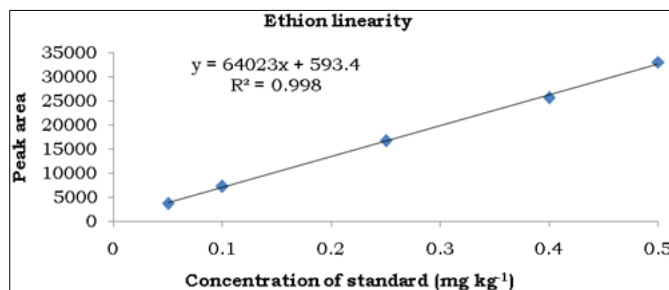
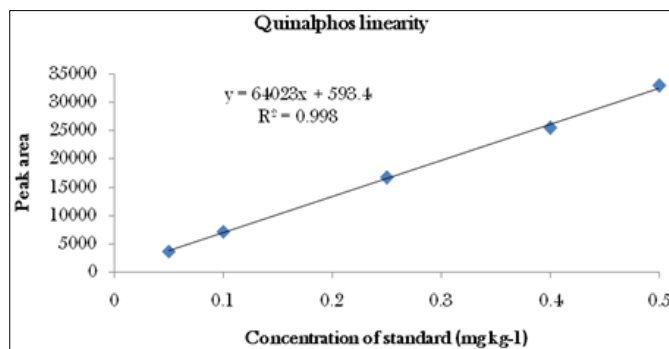
The brinjal samples were extracted and cleaned up using modified QuEChERS method (Quick, Easy, Cheap, Effective, Rugged and Safe (Sharma, 2013) [5]. The entire laboratory sample was crushed thoroughly in grinder and approximately 10 g homogenized sample weighed in a 50 ml polypropylene tube and tube was kept in deep freezer for 10 min. Homogenised sample was extracted with 10 ml ethyl acetate in presence of 10 g anhydrous Na₂SO₄ and centrifuged at 3500 rpm for 5 min. Transfer 2 ml supernatant to the 15 ml tube containing 50 mg PSA. The content was vortexed for 30 Sec and then centrifuged for 2 min at 2500 rpm. The supernatant was filtered through 0.2 micron filter subjected to GC-MS analysis.

Estimation

GCMS Parameters

The analysis of samples for quinalphos and ethion residues was carried out with Shimadzu make GC-MS-QP 2010 plus with auto injector. The operating parameters of the instrument are shown below

Column type Ype	: VF-5MS, 30 m × 0.25 µm × 0.25 mm
Column temperature	: 80 ^o C 1 min hold @ 11 ^o C/min 140 ^o C 3 min hold @ 5 ^o C/min 225 ^o C 5 min hold @ 8 ^o C/min 280 ^o C 7 min hold
Injector temperature	: 250 ^o C
Interface temperature	: 285 ^o C
Ion source temperature	: 250 ^o C
Injection volume	: 1 µl
Column flow	: 1.5 ml min ⁻¹
Retention time approx.	: Quinalphos: 10.60 min Ethion: 12.64 min



Tabal 1: Mean Recovery of different insecticides in brinjal

Fortification level	Recovery (%)	
	Quinalphos 25 EC	Ehion 50 EC
0.05 mg/kg	89.33 (9.27)	107.53(7.94)
0.25 mg/kg	96.83 (5.40)	82.66 (1.25)
0.50 mg/kg	84.90 (1.67)	91.43 (1.15)

Results and Discussion

Persistence and dissipation of quinalphos in/on brinjal fruits

In case of quinalphos 25 EC @ 250 and 500 g a.i.ha⁻¹, initial deposits were recorded as 0.38 and 0.74 mg kg⁻¹ and they are

found to be below detection limit (BDL) on 7th and 10th day, respectively. The half-life (RL₅₀) values of quinalphos for brinjal were 2.05 and 2.18 days, respectively (Table 2). The residues dissipated to 81.57 and 90.54 per cent at both the doses of application (Table 2).

Table 2: Dissipation pattern of quinalphos in brinjal fruits

Interval between last application and sampling	Quinalphos			
	Recommended dose @ 250 g a.i.ha ⁻¹		Double the recommended dose @ 500 g a.i.ha ⁻¹	
	Mean residues (mg kg ⁻¹)	Dissipation (%)	Mean residues (mg kg ⁻¹)	Dissipation (%)
0 day (2 hrs)	0.38	-	0.74	-
1 day	0.25	34.21	0.44	40.54
3 day	0.12	68.42	0.22	70.27
5 day	0.07	81.57	0.15	79.72
7 day	BDL	-	0.07	90.54
10 day	BDL	-	BDL	-
15 day	BDL	-	BDL	-
RL ₅₀ (days)	2.05		2.18	

* BDL= Below Detection Limit *LOD= Limit of detection *LOD Limit of quantification

Present findings are agreement with Pathan *et al.*, (2012) [4] who reported that dissipation of quinalphos in brinjal and soil at 375 and 750 g a.i.ha⁻¹ recommended dose and double the recommended dose. The initial deposits were recorded as 0.09 and 0.15 mg kg⁻¹ for and reached below detection limit in 7th and 10th day at recommended and double the recommended dose, respectively. The half-life values calculated were 2.00 and 3.00 days for recommended and double the recommended

dose, respectively. Similar type of studies were conducted by Mutkule *et al.*, (2014) [3] reported that dissipation of quinalphos in brinjal at recommended dose and double the recommended dose. Initial deposits were dissipated to below detectable limit on 7th and 10th day for both doses, respectively.

Persistence and dissipation of ethion on brinjal fruits

Table 3: Dissipation pattern of ethion in brinjal fruits

Interval between last application and sampling	Ethion			
	Recommended dose @ 500 g a.i. ha ⁻¹		Double the recommended dose @ 1000 g a.i. ha ⁻¹	
	Mean residues (mg kg ⁻¹)	Dissipation (%)	Mean residues (mg kg ⁻¹)	Dissipation (%)
0 day (2 hrs)	0.26	-	0.39	-
1 day	0.16	38.46	0.21	46.15
3 day	0.08	69.23	0.14	64.10
5 day	0.05	80.76	0.12	69.23
7 day	BDL	-	0.07	82.05
10 day	BDL	-	BDL	-
15 day	BDL	-	BDL	-
RL ₅₀ (days)	2.12		3.21	

* BDL= Below Detection Limit *LOD= Limit of detection *LOD Limit of quantification

As regards ethion 50 EC @ 500 and 1000 g a.i. ha⁻¹, initial deposits were recorded as 0.26 and 0.39 mg kg⁻¹. The ethion residues were below detection limit (BDL) on 7th and 10th day. The residual half-life (RL₅₀) values recorded were 2.12 and 3.21 days, respectively (Table 3). The residues of ethion dissipated to 80.76 and 82.02 per cent (Table 3) at recommended and double the recommended dose, respectively.

These results obtained were found to be in agreement with those of Singh *et al.*, (2007) [7] who studied the residues of ethion in cucumber following three applications of the insecticide at 375 and 750 g a.i.ha⁻¹. Residues of ethion dissipated below the maximum residue limit (MRL) of 0.5 mg kg⁻¹ in 7 days. Half-life values (RL₅₀) values were observed in the range of 2.92 days at recommended dose. Varghese *et al.*, (2011) [8] indicated that the dissipation of ethion 50 EC at 375 g a.i.ha⁻¹, on chilli. The residues of the insecticides persisted up to 15th day and reached below detection limit (BDL) on 20th day of spraying and half-life values were calculated as 3.43 days on chilli.

Sharma and Parihar (2013) [6] also reported that dissipation and persistence of ethion 50 EC residues on chilli. Two sprays were given of ethion at 500 and 1000 g a.i.ha⁻¹ at 45th days

after transplanting and second spray at 15 days interval of first spray. The initial deposit of ethion were 2.40 and 4.84 mg kg⁻¹, half-life values were 1.81 and 2.32 days at both recommended and double the recommended dosage.

Conclusion

These pesticides are being used by the farmers for the pest management in brinjal through they are non-recommended. As there is no MRL available for quinalphos, and ethion in brinjal, considering 0.05 mg/kg as MRL, preharvest interval (PHI) of seven days can be suggested for harvesting brinjal fruits free from residues of quinalphos, and ethion.

References

- Anonymous. Indian Horticulture Database 2013. Published by National Horticulture Board, Ministry of Agriculture, and Govt. of India, Agrihort. 2013; 8:1379-1398.
- Khay JH, Choi AM, Abd El-Aty MIR, Mamun BJ, Park A, Goudah HC *et al.* Dissipation behavior of lufenuron, benzoylphenylurea insecticide in/ on chinese cabbage applied by foliar spraying under greenhouse condition. Bull. Environ. Contam Toxicol. 2008; 3:369-372.

3. Mutkule DS, Singh S, Patel ZP, Patel KN, Patel KG. Persistence and dissipation of quinalphos and fipronil applied to brinjal (*Solanum melangena* L.). Bioinfolate, 2014; 11(2):640-645.
4. Pathan ARK, Parihar NS, Sharma BN. Dissipation study of quinalphos in/on brinjal and soil. Bull. Environ. Contam. Toxic. 2012; 88:894-896.
5. Sharma KK. Pesticide Residue Analysis Manual, ICAR, Gov.of India, 2013, 90-91.
6. Sharma BN, Parihar NS. Dissipation and persistence of dimethoate and ethion residues in/on chilli (*Capsicum annum* L.). Pestic. Res. J. 2013; 25(1):80-82.
7. Singh G, Battu RS, Jyot G, Singh B, Joia BS. Persistence of ethion residues on cucumber, *Cucumis sativus* (Linn.) using Gas Chromatography with nitrogen phosphorus detector. Bull. Environ. Contam. Toxicol. 2007; 79:437-439
8. Varghese TS, Mathew TB, George T, Beevi SN, Xavier G. Dissipation study of dimethoate, ethion and oxydemeton methyl in chilli. Pestic. Res. J. 2011; 23(1):68-73.
9. Vevai. Know your crop, its problem and control brinjal pesticide. 1970; 4(4):26-35.