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Dissipation and persistence of Triazophos In/On Brinjal

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Abstract
Field trial was conducted to study the dissipation pattern of triazophos in/on brinjal by following two foliar applications at recommended (500 g a.i./ha) and double the recommended dose (1000 g a.i./ha) at fruit initiation stage. The brinjal crop was sprayed twice at 10 days interval. Initial residues of triazophos in brinjal fruits were recorded as 0.90 and 1.85 mg kg^{-1} on brinjal fruits with the half life of 2.10 and 2.04 days at recommended and double recommended dose, respectively. The residues of triazophos reached below quantification limit (BQL) after 7 and 10 days in both the doses. Considering this, Pre-Harvest Interval (PHI) of seven days can be suggested for triazophos for residue free brinjal.

Keywords: Persistence, triazophos, brinjal.

Introduction
Brinjal or eggplant (*Solanum lycopersicum*) is one of the common vegetable in India. Harvested fruits of brinjal are used for preparation of *Curries*, *Bharatas*, etc. In India, brinjal is cultivated round the year over an area of about 663 thousand ha with a production of 12604 thousand MT (Anon., 2016) [2]. Major brinjal producing states are Orissa, Bihar, Karnataka, West Bengal, Andhra Pradesh, Maharashtra and Uttar Pradesh (Anon., 2014). Brinjal crop is damaged by various insect and non insect pests from nursery stage to harvest.
Farmers rely heavily on pesticides for the control of insect pests. Organophosphates Viz., acephate, profenophos and triazophos are the most common insecticides used by farmers, though these are not recommended in brinjal in India. Recently residues of above insecticides have been reported in different vegetables including brinjal and tomato (Beena Kumari *et al*., 2002; Rao *et al*., 2009) [3, 10]. Their residues are frequently reported in brinjal fruits collected from farm gate and also market. Present investigation was conducted to generate the information on dissipation of triazophos in brinjal by following good agricultural practices. The information would help in fixing the Maximum Residue Limit (MRL) and Post Harvest Interval (PHI) for label claim.

Material and Methods
Field experiment
A supervised field experiment for residue studies was conducted during *Rabi* -2016 at the Instructional Farm, Post Graduate Institute, Mahatma Phule Krishi Vidyapeeth, Rahuri, Dist. Ahmednagar. Brinjal crop was raised by following recommended package of practices. Two sprays were given at an interval of 10 days, initiating the first spray at fruit initiation stage. According to residue studies protocol prescribed by Central Insecticidal Board and Registration Committee (CIB & RC), two doses of triazophos (500 g a.i. ha^{-1} and 1000 g a.i. ha^{-1}) were evaluated for their residues.

Chemicals and Reagents
Certified Reference Material of triazophos of high purity (98.9%) was obtained from Sigma Aldrich and commercial insecticide was purchased from local market of Rahuri. The solvents of HPLC grade were ethyl acetate obtained from Avantor Performance Materials India Limited, Thane (India). PSA and sodium sulphate anhydrous were procured from Agilent Technology, Bangalore and SDFCL, Mumbai, respectively. Working standards were prepared by dissolving reference standards in ethyl acetate.

Residue analysis
Standard preparation
Primary stock solution, intermediate standard and working standards were prepared by dissolving reference standard (Purity – 98.9% and Make- Sigma- Aldrich) in ethyl acetate

Method validation

Prior to analysis of samples, linearity of triazophos was established on GC-FPD. Accuracy and precision of the method was determined by per cent mean recovery and per cent relative standard deviation. Linearity was studied by injecting standard solution of triazophos at five linear concentrations i.e. 0.05, 0.10, 0.25, 0.50 and 1.00 µg g^{-1} in triplicate. The linearity curve was established with concentration of the standard and corresponding peak area. Recovery study was conducted in order to establish the reliability of the method of analysis. The brinjal samples from control plots were used for recovery studies. Ten g homogenized sample was taken in 15 ml polypropylene tube. The samples were spiked with three different concentrations viz. 0.05 (LOQ), 0.25 (5×LOQ) and 0.5 (10×LOQ) µg g^{-1} in triplicate. The extraction and clean up were performed as described earlier. Per cent recovery was calculated by using following formula.

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

Sampling

The brinjal fruit 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 second spray. The collected brinjal samples were brought to the laboratory in polythene bags and processed immediately.

Extraction and clean up

Treated brinjal fruits were extracted by QuEChERS method (Sharma, 2013). The entire laboratory sample (1 Kg) was crushed thoroughly in a mixer cum grinder and approximately 10 g homogenized sample was weighed in a 50 ml polypropylene tube. Tube was kept in the deep freezer for 10 min. Homogenised sample was extracted with 10 ml ethyl acetate in presence of 10 g anhydrous Na_2SO_4 and centrifuged at 3500 rpm for 5 min. Two ml supernatant was transferred to 15 ml tube containing 50 mg PSA. The content was vortexed for 30 sec and then cetrifuged at 2500 rpm for 2 min. The supernatant was filtered through 0.2 micron filter and estimation was done by using Gas chromatography (GC) equipped with FPD. The operating parameters are mentioned in Table 1.

Results and Discussion

Linearity

The results of linearity study are presented in Table 2 and linearity line was drawn. The response of the instrument was linear over the range tested and R^2 value was 0.998 for triazophos (Fig. 1). These results indicated that the GC-FPD analysis is a valid method for residue determination of the tested insecticides in brinjal fruits.

Recovery

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 (2011) [8] and mentioned in Table 3.

Dissipation of triazophos

The results revealed that there was reduction in residue levels of triazophos in/on brinjal with time (Table 4. and Fig. 2). No residues were recorded in any brinjal samples collected from untreated plots. At recommended dose of 500 g a.i. ha^{-1}, mean initial residues of triazophos were 0.90 mg kg^{-1} at two hr after second spray. Initial residues of 0.90 µg g^{-1} further dissipated to 0.63, 0.47, 0.23 and 0.08 mg kg^{-1} at 1, 3, 5 and 7 days, respectively and reached BQL at 10 days.

At double the recommended dose (1000 g a.i. ha^{-1}), mean initial residues of 1.95 mg kg^{-1} dissipated to 1.33, 0.94, 0.46, 0.22 and 0.06 mg kg^{-1} at 1, 3, 5, 7 and 10 days, respectively after second spray. Residues of triazophos dissipated to with a half life of 2.10 and 2.04 days at 500 and 1000 g a.i. ha^{-1}, respectively.

The above findings are in agreement with Pradhan *et al.* (2011) [6] reported initial residues of triazophos in brinjal as 0.49 mg kg^{-1} which dissipated within 10 days. Similarly, Mukherjee *et al.* (2015) [4] registered initial residues of 0.52 mg kg^{-1} in brinjal after application of triazophos 35 EC (combination product) @ 1 lit ha^{-1}. The residues persisted up to 10 days in brinjal after application. The half life calculated was 3.32 days. Studies conducted by Parmar *et al.* (2012) [5] revealed the mean initial residues of 0.54 mg kg^{-1} and persisted up to 7 days with a half life of 1.6 days. As there is no MRL available for triazophos in brinjal, 0.05 µg g^{-1} may be taken as a default MRL. On the basis of this, Pre Harvest Interval (PHI) of seven days can be suggested for triazophos for harvesting brinjal fruits free from residues.

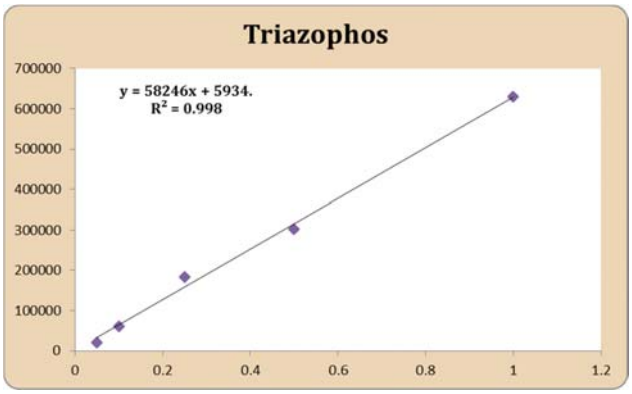

Fig 1: Linearity of triazophos standard

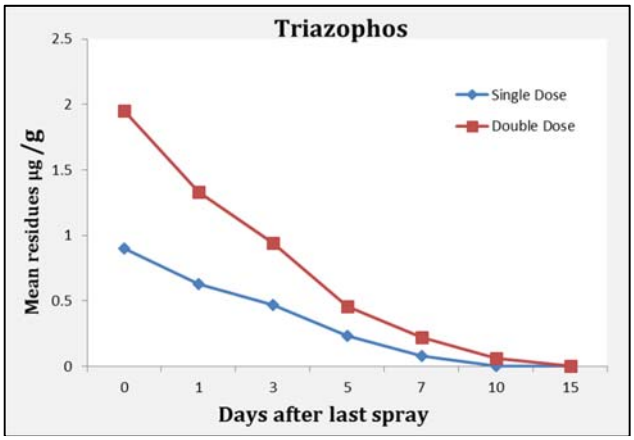

Fig 2: Dissipation pattern of triazophos residues in brinjal

Table 1: Gas chromatographic parameters

Column	DB-1, 30m x 0.25 µm × 0.25 mm
Column Temperature	170^0 C …… 3 min hold @ 6.5^0 C/min 220^0 C …… 2 min hold @ 10^0 C/min 280^0 C …… 6 min hold
Injector Temperature	250^0 C
Column Temperature	170^0 C
Detector Temperature	300^0 C
Injection Volume	1 µl
Column flow	0.96 ml min^{-1}
Hydrogen Flow	90 ml min^{-1}
Air Flow	120 ml min^{-1}

Table 2: Linearity of triazophos standard

Compounds	**Corresponding peak area**				
	0.05 mg/kg	**0.10 mg/kg**	**0.25 mg/kg**	**0.50 mg/kg**	**1.00 mg/kg**
Triazophos	20413	61504	182669	302164	629299

Table 3: Recovery of triazophos in brinjal fruits

Fortification Level (mg/kg)	**Recovery (%)**			
	R-I	**R-II**	**R-III**	**Mean**
0.05	90.42	96.98	98.22	95.21 (±4.40)
0.25	87.32	80.82	85.09	84.41 (±1.77)
0.50	88.22	88.59	88.06	88.63 (±0.47)

Table 4: Residues of triazophos in brinjal fruits

Interval between last application and sampling	**Triazophos**			
	Recommended dose		**Double the recommended dose**	
	Mean Residues (µg g^{-1})	**Dissipation (%)**	**Mean Residues (µg g^{-1})**	**Dissipation (%)**
0 day (2 hr.)	0.90 (± 0.06)	-	1.95 (± 0.05)	-
1 day	0.65 (±0.03)	30.00	1.33 (±0.03)	31.79
3 day	0.47 (±0.03)	47.70	0.94 (±0.04)	51.79
5 day	0.23 (±0.02)	74.44	0.46 (±0.02)	76.41
7 day	0.08 (±0.01)	91.11	0.22 (±0.07)	88.71
10 day	BQL	-	0.06 (±0.02)	96.92
15 day	BQL	-	BQL	-
$RL_{50(days)}$	2.10		2.04	

BQL - Below Quantification Level LOQ - 0.05 mg kg^{-1}

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