Bulletin of Environment, Pharmacology and Life Sciences Bull. Env. Pharmacol. Life Sci., Vol 6 [12] November 2017: 63-66 ©2017 Academy for Environment and Life Sciences, India Online ISSN 2277-1808 Journal's URL:http://www.bepls.com CODEN: BEPLAD Global Impact Factor 0.876 Universal Impact Factor 0.9804 NAAS Rating 4.95 ORIGINAL ARTICLE



Persistence of Acephate, Triazophos And Profenophos In/On Okra

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ABSTRACT

Persistence of acephate, triazophos and profenophos in/on okra was studied following application of insecticide at fruit initiation stage. Acephate @ 560 g ai./ha, triazophos @ 500 g ai./ha and profenophos @ 500 g ai./ha were applied twice at an interval of 10 days. The residues dissipated with half life of 1.88, 1.89 and 1.59 days in case of acephate, triazophos and profenophos, respectively.

KEY WORDS - Profenophos, triazophos, acephate and persistence

Received 04.10.2017

Revised 19.10.2017

Accepted 02.11.2017

INTRODUCTION

Okra, *Abelmoschus esculentus* (L.) Monech, is an important vegetable crop in India. It is infested by several insect pests among which shoot and fruit borer is the most destructive. The average fruit infestations have been estimated to be 35-76 per cent [2]. In order to control the damage caused by insect pests, substantial amounts of insecticides are used by the farmers, even during fruiting stage. Indiscriminate use of insecticides, particularly at fruiting stage and non adoption of safe waiting period leads to accumulation of residues in consumable produce. Organophosphorus insecticides like triazophos, profenophos and acephate though not recommended are being used for the control of insect pests in vegetable crops. Recently residues of above insecticides have been reported in different vegetables including okra. Keeping this in mind, the investigations were undertaken to study the persistence of acephate, triazophos and profenophos in okra.

MATERIAL AND METHODS

Field Experiment

The field experiment was conducted during *Kharif* 2014 at the Instructional Farm, Department of Entomology, Mahatma Phule Krishi Vidyapeeth, Rahuri, Dist. Ahmednagar. Okra crop was raised by following recommended package of practices. Treatments included acephate, triazophos and profenophos in randomized block design.

Test chemicals

The Certified Reference Materials of acephate, triazophos and profenophos were made available by Pesticide Residue Laboratory, AINP on Pesticide Residues, MPKV, Rahuri whereas the commercial formulations were made available by Modern Pesticide Testing Scheme, Mahatma Phule Krishi Vidyapeeth, Rahuri.

Extraction and clean up:

Okra fruits (1 kg) were collected from each replication on 0 (2 hr after application), 1, 3, 5, 7, 10 and 15 days after second application of insecticides and samples were brought to Pesticide Residue Laboratory and processed immediately. Estimation of residues of insecticides was carried out by QuEChERS method [1]. The entire laboratory sample (1 kg) was cut and homogenized in grinder properly. Weighed 10 g homogenized sample in a 50 ml polypropylene tube and tube was kept in deep freezer for 10 min. To this, added 10 ml ethyl acetate and 10 g anhydrous sodium sulphate, hand shaked vigorously and centrifuged the content at 3500 rpm for 5 min. Transferred 2 ml supernatant to the 15 ml tube containing 50 mg PSA.

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The content was vortexes for 30 sec and then centrifuged at 2500 rpm for 2 min. The supernatant was filtered through 0.2 micron filter in GC vials and performed GC-MS analysis using operation parameters as mentioned in Table 1.

Standard Preparation:

An accurately weighed 10 mg of an individual certified reference standard was dissolved in 10 ml volumetric flask using suitable solvent (toluene) to prepare the standard stock solution to 1000 mg/kg. Standard stock solution of each insecticide was further diluted to obtain immediate lower concentrations of 100 and 10 mg/kg. They were stored in a refrigerator at -20°C. From intermediate standards, working standards of 0.50, 0.40, 0.25, 0.10 and 0.05 mg/kg1 were prepared by suitably diluting the stock solution in n-hexane and used as standard check in residue determination, linearity and recovery studies.

Method validation:

Prior to analysis of samples, linearity of acephate, triazophos and profenophos was established on GCMS. Accuracy and precision of the method was determined by per cent mean recovery and per cent relative standard deviation (RSD).

Linearity:

Five linear concentrations (0.05 mg/kg, 0.10 mg/kg, 0.25 mg/kg, 0.40 mg/kg, and 0.50 mg/kg), each of acephate, triazophos and profenophos were injected in triplicate. Linearity curve was established with concentrations of the standard (CRM) and corresponding peak area.

Recovery:

The analytical method for estimation of acephate, triazophos and profenophos residues in okra fruits has been validated by conducting recovery studies using control samples. Ten gram control sample of blended okra fruits was taken in 50 ml centrifuge tubes in three replicates and each were spiked with acephate, triazophos and profenophos separately at required fortification levels *i.e.* 0.05 mg/kg (LOQ), 0.25 mg/kg (5 x LOO) and 0.5 mg/kg (10 x LOO) by adding an appropriate volume of working standard of 10 mg/kg. This mixture was then shaken, in order to attain a proper homogeneity of insecticides in the sample. The extraction and cleanup procedure was followed as described earlier. Per cent recovery was calculated by using following formula.

Quantity of pesticide recovered

Per cent recovery = -× 100

Quantity of pesticide added

RESULTS AND DISCUSSION

Linearity studies

Linearity was studied with pure standard solutions of acephate, triazophos and profenophos at five linear concentrations and mentioned in Table 2. Linearity curve was established with concentrations of the standard and corresponding peak area. Good linearity was achieved and the regression coefficient (R2) was greater than 0.99 over the range tested for all three insecticides (Fig. 1).

Recovery studies

The results of recovery study are presented in Table 3. The recovery of acephate carried out at the levels of 0.05, 0.25 and 0.50 mg/kg in okra fruits was between 89.13 - 101.08 % with standard deviation of 2.04–6.64 %. The results on the recovery of triazophos, carried out at the levels of 0.05, 0.25 and 0.50 mg/kg in okra fruits were between 85.64–118.04 % with standard deviation of 2.29–4.21 %. Whereas the results on the recovery of profenophos carried out at the levels of 0.05, 0.25 and 0.50 mg/kg in okra fruits were between 81.58 -100.72 % with standard deviation of 2.24-2.60 %.

Residues/dissipation of acephate, triazophos and profenophos in/on okra

The initial deposit and subsequent residues of acephate, triazophos and profenophos in/on okra fruits at 0, 1,3, 5, 7, 10 and 15 days after last spray are presented in Table 4. In case of acephate, initial deposit of 2.62 mg/kg gradually dissipated to 1.90, 1.03, 0.44 and 0.20 mg/kg at 1, 3, 5 and 7 days, respectively. The half-life (RL50) of acephate was worked out to be 1.88 days.

As regards triazophos, initial deposit of 0.31 mg/kg gradually dissipated to 0.23, 0.11, and 0.05 mg/kg at 1, 3, and 5 days, respectively. The half-life (RL_{50}) of triazophos was worked out to be 1.89 days. In case of profenophos, initial deposit of 4.39 mg/kg gradually dissipated to 2.63, 1.50, 0.76, 0.17 and 0.06 mg/kg at 1, 3, 5, 7 and 10 days, respectively. The half-life (RL_{50}) of profenophos was worked out to be 1.59 days.

The dissipation of pesticide residues in plants depends on the climatic conditions, type of application, dosage and interval between application and time of harvest. The results revealed reduction in residue levels of all these insecticides in okra fruits with time. In case of acephate, the initial deposit of 2.62 mg kg-1 dissipated to BDL on 10th day after second spray. The half life value was 1.88 days at the recommended dose of 560 g a.i/ha. The rate of dissipation was 44-45 per cent with acephate within a day as reported by Prasad et al. [3].

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As regards triazophos, initial deposit 0.31 mg/kg dissipated to BDL on 7th day after second spray. The half life value recorded was 1.89 days. Paras Nath *et al.* [3] recorded 86.2 per cent dissipation of triazophos in okra fruits. Prasad *et al.* [4] reported 62-72 per cent dissipation of triazophos within a day. These reports lend support to present finding. However higher initial deposits of triazophos were detected on okra fruits [5] and rate of dissipation was slow but residue reached BDL after 15 days.

In case of profenophos, initial deposit of 4.39 mg/kg declined to BDL on 15th day. The half life value recorded was 1.59 days. In okra fruits, dissipation of profenophos was more than 98.4 % as recorded by Paras Nath et al. 2005 which tally with the present investigation for profenophos. It may be inferred that, by considering the BQL i.e. 0.05 mg/kg as safe value for all three insecticides, seven days, ten days and fifteen days may be considered safe in case of triazophos, acephate and profenophos for harvesting residue free okra fruits.

The data generated on persistence of acephate, triazophos and profenophos in/on okra will be useful for the determination of safe waiting period of these non recommended insecticides.

Column Type	VF-5-MS-30m x 0.25 μm × 0.25 mm
Column Temperature	80° C 1 min hold @ 11° C/min 140° C3 min hold @ 5° C/min 225° C 5 min hold @ 8° C/min 280° C 7 min hold
Injector Temperature	250º C
Interface Temperature	285º C
Ion source Temperature	250º C
Injection Volume	1 ul
Column flow	0.96 ml min ⁻¹

Table 1. GC MS Parameters

Insecticide	Retention	Corresponding Peak Area				
	Time (min.)	0.05 mg/kg	0.10 mg/kg	0.20 mg/kg	0.40 mg/kg	0.50 mg/kg
Acephate	9.95	852	1554	3932	7860	8336
Profenophos	25.78	8395	16944	41261	8100	89000
Triazophos	28.37	18362	38080	74508	15490	181182

Table 3. Recovery of acephate, triazophos and profenophos in okra

Insecticide	Recovery (%)				
	Acephate Profenophos		Triazophos		
	Mean <u>+</u> SD	Mean <u>+</u> SD	Mean <u>+</u> SD		
0.05 mg/kg	90.63 <u>+</u> 2.04	88.10 <u>+</u> 2.29	98.27 <u>+</u> 2.38		
0.25 mg/kg	94.62 <u>+</u> 6.64	16.44 <u>+</u> 1.73	84.55 <u>+</u> 2.60		
0.5 mg/kg	98.25 <u>+</u> 3.41	98.93 <u>+</u> 4.21	93.50 <u>+</u> 2.24		

Table 4. Residues of profenophos in brinjal fruits

Interval between last application	Residues (mg/kg) (Mean of three replications)				
and sampling	Control	Acephate 560 g a.i./ha	Triazophos 500 g a.i./ha	Profenophos 500 g a.i./ha	
0 day (2hr)	ND	2.62	0.31	4.39	
1 day	ND	1.90	0.23	2.63	
3 day	ND	1.03	0.11	1.50	
5 day	ND	0.44	0.05	0.76	
7 day	ND	0.20	BDL	0.17	
10 day	ND	BDL	BDL	0.06	
15 day	ND	BDL	BDL	BDL	
RL50(days)	-	1.88	1.89	1.59	

 * ND - Not Detected BQL - Below Quantification Level LOQ - 0.05 $\mu g\, {\rm g}^{\rm -1}$

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Fig.1 Linearity curve of Acephate, Profenophos and Triazophos

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