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Dissipation and persistence of certain organophosphorus insecticides in/on Okra

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Abstract
Persistence of some organophosphorus insecticides *viz.* acephate, triazophos and profenophos in okra was studied following two applications at recommended dose. Residues persisted upto 5, 7, and 10 days for triazophos, acephate and profenophos, respectively. Half-life values were calculated from first-order dissipation kinetics. In case of acephate, residues on okra fruits dissipated with half-life of 1.88 days, whereas residues of triazophos and profenophos dissipated with the half-life of 1.89 and 1.59 days, respectively. Considering 0.05 mg/kg as LOQ, 7, 10 and 15 day can be considered safe for harvesting residue free okra fruits in case of triazophos, acephate and profenophos, respectively.

Keywords: Okra, organophosphorus insecticides, acephate, profenophos, triazophos, residues

1. Introduction
Okra (*Abelmoschus esculentus* L.) is an important vegetable crop commonly known as bhendi or lady's finger and belongs to family Malvaceae. It is cultivated throughout India with a major share in state of Maharashtra, West Bengal, Uttarpradesh, Karnataka, Gujrat and Madhya Pradesh (Shinde *et al*. 2007) [14]. One of the important limiting factors in cultivation of okra is the damage caused by insect pests. The control of numerous insect pests on this crop envisages use of different insecticides as a traditional and normal practice (Singh *et al*. 2004; Sinha and Sharma, 2007) [16, 17]. In order to combat the insect pest problem, lots of pesticides are used by the vegetable growers. Indiscriminate use of insecticides/pesticides particularly at fruiting stage and non adoption of safe waiting period however, leads to accumulation of pesticide residues in consumable vegetables. Contamination of vegetables with pesticide residues has been reported by several researchers (Kumari *et al*. 2002 and Kumari *et al*. 2003) [5, 6]. Recently residues of above insecticides have been reported in different vegetables including brinjal and tomato (Singh and Gupta, 2002) [15]. In some cases, the residues of these insecticides exceeded their tolerance level. The degradation or dissipation of insecticide is influenced by climatic conditions, type of application, plant species, dosage interval between application and time of harvest (Khay, *et al*., 2008) [4]. It is necessary to determine the dissipation pattern of these commonly used insecticides in okra by following Good Agricultural Practices (GAP). Keeping this in view an attempt was made to study the persistence of acephate, profenophos and triazophos in/on okra.

2. Materials and Methods
2.1 Field experiment
A supervised field experiment for residue studies was conducted during *Rabi* -2014 at the Instructional Farm, Post Graduate Institute, Mahatma Phule Krishi Vidyapeeth, Rahuri, Dist. Ahmednagar. Okra crop was raised by following recommended package of practices and sprayed with acephate 75 SP, triazophos 40 EC and profenophos 50 EC. Acephate 75 SP was sprayed at recommended dose of 560 g. a.i./ha whereas triazophos 40 EC and profenophos 50 EC were sprayed at recommended dose of 500 g. a.i./ha. Two sprays of each insecticide were given separately at an interval of 10 days, initiating the first spray at fruit initiation stage.

2.2 Chemicals and Reagents
Certified Reference Material of acephate, triazophos and profenophos with purity of 97.8, 98.9 and 98.6 percent were obtained from Sigma Aldrich and commercial formulations were 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.

2.3 Residue analysis

2.3.1 Standard preparation: An accurately weighed 10 mg of an individual standard was dissolved in 10 ml volumetric flask using toluene to prepare the standard stock solution of 1000 mg kg-1. Standard stock solution of each insecticide was serially diluted to obtain intermediate lower concentration of 100 mg kg-1. They were stored in a refrigerator at -40ºC. From intermediate standards, working standards were prepared by suitably diluting the stock solution in n-hexane and used as standard check in analysis, linearity and recovery studies.

2.3.2 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 percent mean recovery and percent relative standard deviation. Linearity was studied by injecting standard solution of insecticides under study at five linear concentrations i.e. 0.05, 0.10, 0.25, 0.40 and 0.50 mg/kg. 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 okra 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 mg/kg (LOQ), 0.25 mg/kg (5×LOQ) and 0.5 mg/kg (10×LOQ) in triplicate. The extraction and clean up were performed as described hereunder. Percent recovery was calculated by using following formula.

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

2.3.3 Sampling : The okra 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 samples were brought to the laboratory in polythene bags and processed immediately.

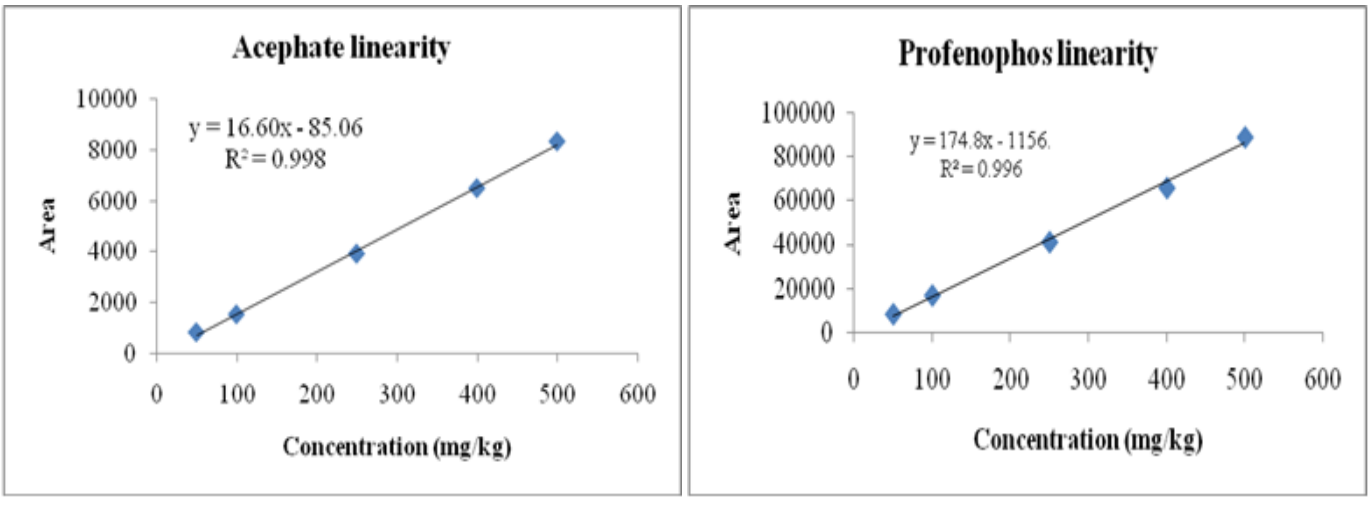
2.3.4 Extraction and clean up: Treated okra fruits were extracted by QuEChERS method (Sharma, 2013) [13]. The entire laboratory sample (1 Kg) was crushed thoroughly in a mixer cum grinder and approximately 10 g homogenized sample was weighed in a 50 ml polypropylene tube. Tube was kept in the deep freezer for 10 min. Homogenized 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 GCMS. The operating parameters were as below

Column	:	VF 5 MS 30m X 0.25 u X 0.25 u
Column Temperature	:	80^0C …… 1 min hold @ 11^0C/min 140^0C ……3 min hold @ 5^0C/min 225^0C ……5 min hold @ 8^0C/min 280^0C ……7 min hold 170º C…….3 min hold @ 6.5 º C/min 220º C…..…2 min hold @ 10 º C/min 280 º C……..6 min hold
Injector temperature	:	250^0 C
Interface temperature	:	285^0C
Ion source temperature	:	220^0 C
Injection volume	:	1 µl
Column flow	:	1.50 ml/min
Retention time Approx	:	Acephate –9.95 min., Profenophos – 25.80 min. Triazophos -28.42 min.

3. Results and Discussion

3.1 Method validation

The detector response to the neat standards of the insecticides was studied by injecting five linear concentrations of different insecticides. The graph was plotted with detector response against respective concentrations and linearity line was drawn. The response of the instrument was linear over the range tested and R^2 value was 0.99 for all the three insecticides (Fig. 1). These results indicated that the GCMS analysis is a valid method for residue determination of the tested insecticides in okra fruits. Accuracy of the analytical method was determined by recovery studies. The percent recovery was within acceptable range of 70-120 percent prescribed by SANCO (2011) [12] and mentioned in Table 1.



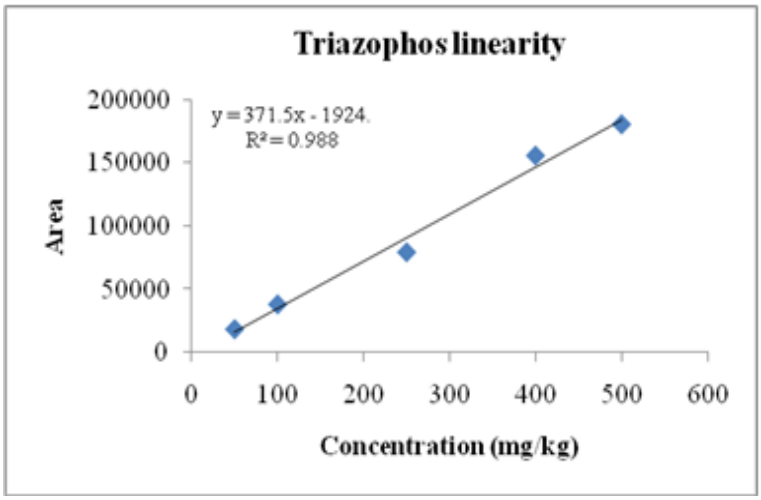


Fig 1: Linearity of acephate, profenophos and triazophos standard

Table 1: Recovery of different insecticides in okra

Fortification level	Recovery (%)		
	Acephate 75 SP	Triazophos 40 EC	Profenophos 50 EC
0.05 mg/kg	89.28 (±0.41)	92.02 (±1.05)	96.59 (±5.71)
0.25 mg/kg	90.81(±2.59)	105.34 (±1.01)	91.04 (±4.52)
0.50 mg/kg	94.52 (±1.72)	91.29 (±1.32)	96.37 (±1.68)

3.2 Dissipation

The results revealed that there was reduction in residue levels of all the three insecticides in/on okra with time (Table 2). No residues were recorded in any okra samples collected from untreated plots. At recommended dose of 560 g a.i. ha^{-1}, mean initial residues of acephate were 2.61 mg/kg at two hr after second spray. Initial residues of 2.61 mg/kg further dissipated to 1.90, 1.03, 0.44 and 0.20 mg/kg with a reduction of 27.20, 60.54, 83.14 and 92.34 percent at 1, 3, 5 and 7 days, respectively. The residues of acephate reached BQL on 10^{th} day of its treatment. Reported the high initial residues (8.44 mg/kg) of acephate when sprayed at recommended dose of 560 g a.i. ha^{-1} in okra.

In case of triazophos, mean initial residues of 0.30 mg/kg dissipated to 0.23, 0.11 and 0.05 mg/kg with a reduction of 23.33, 63.33 and 83.33 percent at 1, 3 and 5 days, respectively. The residues of triazophos reached BQL on 7^{th} day of its treatment. Comparatively higher initial deposits of 2.54 and 6.61 mg/kg were recorded on okra when sprayed with triazophos @ 350 and 700 g a.i./ha as reported by Raj *et al.* (1999) [10] which dissipated with a half-life of 1 day. On the contrary, low initial residues (0.36 mg/kg) were recorded in brinjal by Reddy *et al.* (2007) [11]. Banerji *et al.* (2008) [1] reported the initial residues of triazophos as 0.31 mg/kg when sprayed @ 0.06% a.i./ha in bitter gourd. The dissipation pattern of triazophos with a half life of 1.89 days as observed in present investigation is more less similar to 1.7 days as half life for triazophos at higher dose of 700 g used in cauliflower (Chahal *et al*, 2000) [2].

Mean initial residues (4.39 mg/kg) of profenophos dissipated to 2.63, 1.50, 0.76, 0.17 and 0.06 mg/kg with a reduction of 40.09, 65.83, 82.69, 96.13 and 98.63 percent at 1, 3, 5, 7 and 10 days, respectively. The residues of profenophos reached BQL on 15^{th} day of its treatment. The above findings are in corroboration with Radwan *et al.* (2004) [9] who recorded initial residues of profenophos as 4.50 mg/kg in brinjal. Further, Nigam *et al.* (2009) [7] obtained initial residues of 3.89 mg kg^{-1} when profenophos was applied @ 800 g a.i. ha^{-1} in brinjal. The present findings are in close agreement with Parmar *et al* (2012) [8] in okra & Gupta *et al* (2011) [3] in tomato.

Table 2: Residues of different insecticides in okra

Parameters	Control	Acephate 75 SP 560 g a.i./ha	Percent dissipation	Triazophos 40 EC 500 g a.i./ha	Percent dissipation	Profenophos 50 EC 500 g a.i./ha	Percent dissipation
0 day	ND	2.61(±0.11)	--	0.30(±0.01)	--	4.39(±0.18)	--
1 day	ND	1.90(±0.05)	27.20	0.23(±0.03)	23.33	2.63(±0.18)	40.09
3 day	ND	1.03(±0.08)	60.54	0.11(±0.01)	63.33	1.50(±0.27)	65.83
5 day	ND	0.44(±0.06)	83.14	0.05(±0.01)	83.33	0.76(±0.09)	82.69
7 day	ND	0.20(±0.02)	92.34	BQL		0.17(±0.04)	96.13
10 day	ND	BQL		BQL		0.06 (±0.01)	98.633
15 day	ND	BQL		BQL		BQL	
$DT_{50\ (days)}$		1.88, 1.55		1.89		1.59	

* ND-Not Detected, BQL= Below Quantitation Limit LOQ 0.05 ppm

4. Conclusion

At present, there is no MRL available for acephate, triazophos and profenophos in okra. It may be inferred that, considering 0.05 mg/kg as MRL, pre harvest interval (PHI) of ten, seven and fifteen days can be suggested for acephate, triazophos and profenophos, respectively for harvesting okra fruits free from residues.

5. Acknowledgement

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