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Dissipation and persistence of acephate, triazophos and profenophos in/on brinjal

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Abstract
Studies on the dissipation of acephate, profenophos and triazophos in/on brinjal was undertaken by following two foliar applications at recommended dose (500 g a.i./ha) at fruiting stage. Residues of acephate, profenophos and triazophos dissipated with a half-life of 2.13, 2.64 and 1.97 days, respectively. The residues reached below quantification limit (BQL) on 10th day in all the three insecticides. Considering this, Pre-Harvest Interval (PHI) of ten days can be suggested for acephate, profenophos and triazophos with reduced risk of insecticide residues in brinjal.

Keywords: acephate, profenophos, triazophos, residue, persistence and QuEChERS

Introduction
Brinjal (*Solanum lycopersicon*) 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 692 thousand ha with a production of 12634 thousand MT (Anon., 2014) [3]. Orissa, Bihar, Karnataka, West Bengal, Andhra Pradesh, Maharashtra and Uttar Pradesh are the major brinjal producing states in the country (Anon., 2014) [3]. Brinjal crop is damaged by various insect pests *i.e.* shoot and fruit borer (*Leucinodes orbnalis* G.), whitefly (*Bemisia tabaci* G.) and leaf hopper (*Amrasca devanstans* D.) (Butani and Jotwani, 1984) [6]. of these, shoot and fruit borer (*Leucinodes orbnalis* G.) is the most serious insect pest of brinjal. The average crop losses due to this pest were estimated to be 11 to 93 per cent (Rai *et al*., 2014) [15].
Pesticides, as a key component of integrated pest management, played an important role in increasing agricultural production, but their misuse has led to the environmental pollution including health hazards (Akbar *et al*., 2010) [1]. In order to control insect pests, farmers rely heavily on pesticides. Sometimes pesticides are applied during fruiting stage. Indiscriminate use of pesticides has resulted in the accumulation of pesticide residues in the primary agricultural products as well as soil (Baig *et al.*, 2009) [4]. Recently residues of some organophosphorus insecticides have been reported in different vegetables including brinjal and tomato (Kumari B *et al*, 2002; Singh and Gupta, 2002) [9, 20]. 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) [8]. It is necessary to determine the dissipation pattern of commonly used op insecticides in brinjal by following Good Agricultural Practices (GAP). Keeping this in view an attempt was made to study the persistence of acephate, triazophos and profenophos in/on brinjal.

Material and Methods
Field experiment
Residues and dissipation of acephate, triazophos and profenophos in brinal was studied by conducting supervised field experiment during *Rabi*-2014 at the Instructional Farm, Post Graduate Institute, Mahatma Phule Krishi Vidyapeeth, Rahuri, Dist. Ahmednagar. Brinjal seedlings were transplanted to raise the healthy crop by following recommended package of practices. The crop was sprayed with acephate 75 SP @ 560 g. a.i./ha, triazophos 40 EC and profenophos 50 EC @ 500 g. a.i./ha. Two sprays of each insecticide were given separately at an interval of 10 days, initiating the first spray at fruiting stage.

Chemicals and Reagents
Certified Reference Material of acephate, triazophos and profenophos with purity of 97.8, 98.9 and 98.6 per cent were obtained from Sigma Aldrich and commercial formulations were purchased from local market of Rahuri. HPLC grade ethyl acetate was 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

An accurately weighed 10 mg of an individual standatd 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.

Method validation

Prior to analysis of samples, linearity of acephate, triazophos and profenophos 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 insecticides under study 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. For this purpose, brinjal samples from control plots were used. 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) mg/kg. The extraction and clean up were performed as described earlier. Per cent recovery was calculated by using following formula.

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

Sample collection

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) [19]. 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 Gas chromatography (GC) equipped with FPD. The operating parameters are as below.

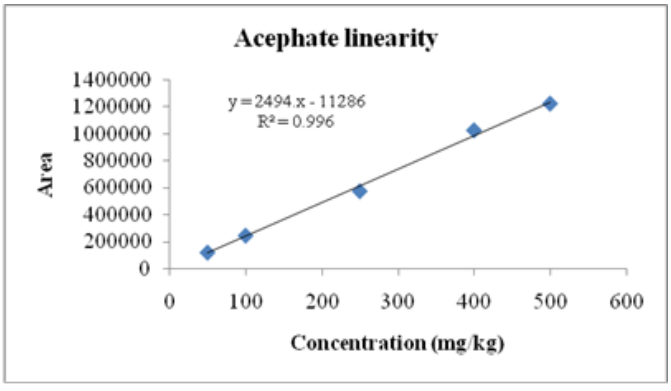
Column	:	Varian DB-5 30m X 0.25 u X 0.25 u
Column Temperature	:	170º C…….3 min hold @ 6.5 º C/min 220º C…..…2 min hold @ 10 º C/min 280 º C……..6 min hold
Injector temperature	:	250 º C
Column temperature	:	170 º C
Detector temperature	:	300 º C
Injection volume	:	1 µl
Column flow	:	0.96 ml/min
Hydrogen flow	:	90 ml/min
Air flow	:	120 ml/min
Retention time Approx	:	Acephate –3.84 min., Profenophos – 13.22 min. Triazophos -15.21 min.

Results and Discussion

The detector response to the neat standards of the insecticides was studied by injecting five linear concentrations of the 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 GC-FPD 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 per cent recovery was within acceptable range of 70-120 per cent prescribed by SANCO (2011) [18] and mentioned in Table 1.

Table 1: Recovery of different insecticides in brinjal

Fortification level	Recovery (%)		
	Acephate 75 SP	Triazophos 40 EC	Profenophos 50 EC
0.05 mg/kg	76.95 (6.30)	97.06 (2.87)	98.55 (4.18)
0.25 mg/kg	100.57(5.5)	114.81(2.5)	77.93(3.5)
0.50 mg/kg	91.79 (3.8)	85.44 (1.6)	88.67 (4.7)



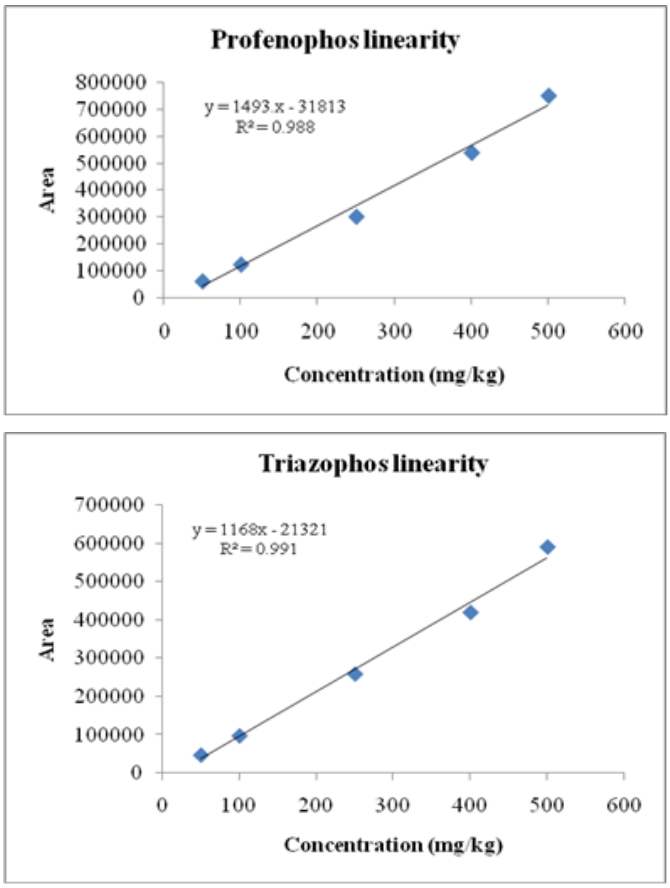


Fig 1: Linearity of acephate, profenophos and triazophos standard

Dissipation of insecticides

The results revealed that there was reduction in residue levels of all the three insecticides in/on brinjal with time (Table 2). No residues were recorded in any brinjal samples collected from untreated plots. At recommended dose of 560 g a.i. ha^{-1}, mean initial residues of acephate were 2.52 mg/kg at two hr after second spray which further dissipated to 1.53, 1.06, 0.44 and 0.25 mg/kg at 1, 3, 5 and 7 days, respectively and reached BQL at 10 days. Per cent reduction in residues was 39.37, 57.86, 82.96 and 90.22 per cent on above days. The half life recorded was 2.14 days. The present findings are in agreement with Patel *et al* (2015) [12] who reported initial residues of 2.43 mg/kg which persisted upto 7 days with a half life of 2.20 days. According to Eijaza *et al* (2015) [7] the half life of acephate was calculated as 2.5 days in okra. Soudamini *et al.* (2016) [21] 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 1.40 mg/kg dissipated to 1.20, 0.79, 0.23 and 0.14 mg/kg at 1, 3, 5 and 7 days, respectively after second spray. The per cent reduction in residues was 13.84, 43.20, 83.53 and 90.21 per cent on above days. Raj *et al*. (1999) [16] reported comparatively low initial deposits of 0.165 and 0.218 mg/kg on brinjal when sprayed with triazophos @ 350 and 700 g a.i./ha which dissipated with a half-life of 1 and 2 days for both the doses. Banerjee *et al*. (2008) [5] reported low initial residues of triazophos as 0.31 mg/kg when sprayed @ 0.06% a.i./ha in bitter gourd. The present finding is in close agreement with Patel *et al* (2015) [12] who recorded initial residues of 1.317 mg/kg in triazophos. The half life recorded was 2.85 days. The differences in half life values could be attributed to formulations and agroclimatic conditions.

Mean initial residues (0.56 mg/kg) of profenophos dissipated to 0.39, 0.26, 0.13 and 0.09 mg/kg at 1, 3, 5 and 7 days, respectively and reached BQL at 10 days. The per cent reduction in residues was 30.54, 52.69, 76.05 and 84.43 per cent on above days, respectively. The above findings are in corroboration with Mukherjee *et al.* (2012) [10] who recorded initial residues of profenophos as 0.575 mg kg^{-1} in brinjal and residues dissipated within 7 days to BQL. Similarly, Reddy *et al.* (2007) [17] determined dissipation of profenophos in chilli and recorded initial residues of 0.36 mg/kg. On the contrary, higher initial residues (4.50 µg g^{-1}) of profenophos were recorded in brinjal by Radwan *et al.* (2004) [13]. Further, Nigam *et al.* (2009) [11] obtained initial residues of 3.89 mg kg^{-1} when profenophos was applied @ 800 g a.i. ha^{-1} in brinjal. Radwan *et al* (2005) [14] recorded half life of 1.96 days in brinjal.

Table 2: Dissipation of acephate, triazophos and profenophos in brinjal

Parameters	Control	Acephate 75 SP 560 g a.i./ha	Per cent dissipation	Triazophos 40 EC 500 g a.i./ha	Per cent dissipation	Profenophos 50 EC 500 g a.i./ha	Per cent dissipation
0 day	ND	*2.52 (±0.16)	--	1.40 (±0.05)	--	0.56 (±0.04)	--
1 day	ND	1.53 (±0.21)	39.37	1.20 (±0.06)	13.84	0.39 (±0.05)	30.54
3 day	ND	1.06 (±0.16)	57.86	0.79 (±0.06)	43.20	0.26 (±0.02)	52.69
5 day	ND	0.44 (±0.12)	82.69	0.23 (±0.04)	83.53	0.13 (±0.01)	76.05
7 day	ND	0.25 (±0.05)	90.22	0.14 (±0.03)	90.21	0.09 (±0.02)	84.43
10 day	ND	BQL		BQL		BQL	
15 day	ND	BQL		BQL		BQL	
$DT_{50\ (days)}$		2.13 2.12		1.97		2.64	

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

• Mean of three replicates

Figures in parenthesis are SD values.

Conclusion

These pesticides are being used by the farmers for the pest management in brinjal though they are non recommended. As there is no MRL available for acephate, triazophos and profenophos in brinjal, considering 0.05 mg/kg as a MRL, pre harvest interval (PHI) of ten days can be suggested for acephate, triazophos and profenophos for harvesting brinjal fruits free from residues.

References

1. Akbar MF, Muhammad AH, Farzana P, Nikhat Y, Sayeed SA. Determination of synthetic and bio-insecticides residues during aphid *(Myzus persicae* (Sulzer) control on cabbage crop through high performance liquid chromatography. Pak. Entomol., 2010; 32(2):156-162.

2. Anastassiades M, Lehotay SJ, Stajnbaher D, Schenck FJ. Fast and easy multiresidue method employing acetonitrile extraction/partitioning and dispersive solid phase extraction for the determination of pesticide residue in produce. J AOAC Int. 2003; 86:412-431.
3. Anonymous. Indian Horticulture Database, National Horticulture Board and Ministry of Agriculture, Govt. of India, 2014, 131.
4. Baig SA, Akhtera NA, Ashfaq M, Rafique MA. Determination of the organophosphorus pesticide in vegetables by high performance liquid chromatography. American Eurasian. J Agric. Envir. Sci. 2009; 6(5):513-519.
5. Banerji R, Sahoo SK, Jha S, Banerjee H. Persistence and dissipation of triazophos in bitter gourd. J Hortl Sci. 2008; 3(1):82-83.
6. Butani DK, Jotwani MG. Insects in vegetables. Periodical Export Book Agency, New Delhi. 1984, 5-7.
7. Eijaza S, Khan MF, Mahmood K, Mohammad A, Alamgir A, Khatri I. Studies on Degradation and Efficacy of Synthetic Pesticides on Okra Crop. Academic Journal of Entomology. 2015; 8(1):12-18.
8. Khay JH, Choi AM, Abd El-Aty MIR, Mamun BJ, Park A, Goudah HC, *et al*. Dissipation behavior of lufenuron, benzoyl phenyl urea insecticide in/ on chinese cabbage applied by foliar spraying under greenhouse condition. Bull. Envir. Contamin. Toxicol. 2008; 3:369-372.
9. Kumari B, Madan VK, Kumar R, Kathpal TS. Monitoring of seasonal vegetables for pesticide residues. Envir. Monit. Assessm. 2002; 74:263-270.
10. Mukherjee I, Kumar A, Kumar A. Persistence behavior of combination mix crop protection agents in/on eggplant fruits. Bull. Envir. Contamin. Toxicol. 2012; 88:338-343.
11. Nigam RC, Pandey RK, Tiwari DD, Katiyar NK. Persistence of endosulfan and profenophos in/ on brinjal. Pesticide Res. J. 2009; 2(2):180-182.
12. Patel DJ, Parmar KD, Joshi MN, Shah PG, Pater AR. Residues and persistence of acephate, profenophos and triazophos in/on brinjal. Journal of Insect Science. 2015; 28(1):99-102.
13. Radwan MA, Shibbob MH, Abu-Elamayem MM, Abdel AA. Residues of pirimiphos - methyl and profenophos on green pepper and egg plant fruits and their effect on some quality properties. Emir. J Agric. Sci. 2004; 16(1):32-42.
14. Radwan MA, Abu-Elamayem MM, Shibbob MH, Abdel AA. Residual behavior of profenophos on some field grown vegetables and its removal using various washing solutions and household processing. Fd. Chem. Toxicol. 2005; 43:553-557.
15. Rai AB, Halder J, Kodandaram MH. Emerging insect pest problems in vegetable crops and their management in India: An appraisal. Pest Mgmt. Hort. Ecosyst. 2014; 20(20):113-122.
16. Raj MF, Shah PG, Patel BK, Patel BA, Patel JA. Dissipation of Triazophos from Brinjal and Okra fruits. Pesticide Research Journal. 1999; 11(1):102-105.
17. Reddy KD, Reddy KN, Mahalingappa PB. Dissipation of fipronil and profenophos residues in chillies (*Capsicum annuum* L.). Pesticide Res. J. 2007; 19(1):106-107.
18. SANCO. Method Validation and quality control procedures for pesticide residue analysis in food and feed. Document No. 2011; 8:15.
19. Sharma KK. Pesticide Residue Analysis Manual, ICAR, Gov. of India, 2013, 90-91.
20. Singh B, Gupta A. Monitoring of pesticide residues in farm gate and market samples of vegetables in a semiarid, irrigated area. Bull. Envir. Contamin. Toxicol. 2002; 68(5):747-751.
21. Soudamini M, Gourishankar M, Siddamallaiah L, Buddidhathi R, Matadha NY. Dissipation of acephate and methamidophos residues on brinjal (*Solanum melongena* L.) and okra (*Abelmoschus esculentus* L.). Pol J Environ Stud. 2017; 26(3):1165-1172.