

PERSISTENCE OF ACEPHATE, PROFENOFOS AND TRIAZOPHOS RESIDUES IN BRINJAL FRUITS AND SOIL

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ABSTRACT

The persistence pattern of acephate, profenofos and triazophos in brinjal fruits was studied after spraying the crop twice at 10 days interval at recommended rate (RR) and double recommended rate (DRR). Acephate applied at the rate 560 g a.i. ha⁻¹ and 1120 g a.i. ha⁻¹. Application rate of profenofos and triazophos were @ 500 g a.i. ha⁻¹ and 1000 g a.i. ha⁻¹. Initial deposits of acephate in brinjal fruits from the two treatments were resulted in 0.683 and 1.080 mg kg⁻¹ on brinjal fruits with the half life of 1.8 and 2.2 days. Initial deposits of profenofos in brinjal fruits from the two treatments were leads to 1.966 and 2.460 mg kg⁻¹ on brinjal fruits which reduced to half in 1.5 and 1.9 days. Initial deposits of triazophos residues were 1.100 and 2.233 mg kg⁻¹ at 500 and 1000 g a.i. ha⁻¹ dissipated to half in 1.6 and 1.9 days. In soil, residues of acephate persisted for 3-5 days, where as residues of profenofos and triazophos persisted for 5-10 days only. Based on the persistence studies waiting period of 9.4 day is suggested for acephate and 8 days for profenofos and triazophos on brinjal from consumer's safety point of view.

INTRODUCTION

Brinjal (*Solanum melongena* L.) is a popular solanaceous and economically important vegetable and is native to India. After potato, it ranks second highest consumed vegetable in India. Annually, India produced about 11.896 million MT of brinjal, on 6.8 lakh hectare of land in different parts of the country. India is the second largest producer of brinjal after China with a 27.55 per cent world production share. It contributes 8.1 per cent of the total vegetable production of the country (Anonymous, 2011). Brinjal is attacked by a number of insect pests from seedling to fruiting stage affecting its growth and productivity. Patial and Mehta (2008) reported 27 insect-pest species attacking this crop. Major insect-pests of brinjal are fruit and shoot borer *Leucinodes orbonalis*, beetles *Henosepilachna vigintioctopunctata*, jassids *Amrasca devastans*, brinjal stem borer *Euzophera perticella* and aphids *Aphis gossypii* (Walia et al., 2010). Fruit and shoot borer (*Leucinodes orbonalis*) is the most devastating pest causing a significant yield loss of 60-70 per cent in India (Kumar et al., 2011). This crop is sprayed heavily with insecticides to protect it from the attack of pests. Different insecticides are approved and recommended against insect-pests of various crops. However, the recommended use of these insecticides vis-à-vis their effectiveness on wide range of pests, provoke the farmers to use them against pests of other crops also, on which they are not recommended. Improper and injudicious use of pesticide, besides posing health threat to the farm workers, also leave harmful pesticide residues on the crop and soil and causes development of pest resistance leading to the losses to the crops (Kumar and Singh, 2014). An extensive study in

India conducted under the project 'Monitoring of Pesticide Residues at National Level' revealed the presence of non approved acephate, profenofos and triazophos residues in brinjal fruits throughout the country. Due to the presence of harmful residues of these pesticides in vegetables. The persistence of acephate, profenofos and triazophos has been studied on various commodities by different workers (Mohapatra et al., 2011; Kaur et al., 2015; Banshtu et al., 2015; Nigam et al., 2009; Shashi et al., 2015; Singh et al., 2015). In order to recommend a safe waiting period an attempt was made to generate persistence data according to good agricultural practices (GAP) for fixation of MRL of these insecticides on brinjal crop by the FSSAI. The present investigation was carried out to study the persistence behaviour of acephate, profenofos and triazophos residues in brinjal fruits soil.

MATERIALS AND METHODS

The experiment was laid out in randomized block design at the experimental farm of the Department of Entomology, Dr. Yashwant Singh Parmar university of Horticulture and Forestry Nauni, Solan (H.P.) during 2013. Brinjal (*Solanum melongena* L.) variety Pusa Purple Long was planted at planting distance of 60×45 cm, as per standard package of practices in 3×2 sq. m plot (Anonymous, 2009).

Acephate (Acevip 75SP), Profenofos (Profex 50 EC) and Triazophos (Triazophos 40 EC) were purchased from local market. The brinjal crop was sprayed twice at 10 days fruit formation stage. Acephate, profenofos and triazophos insecticides were sprayed on brinjal crop at the recommended

rate (RR) and double recommended rate (DRR) using Knapsack sprayer fitted with hollow cone nozzle. Acephate was sprayed at the rate 560 g a.i. ha⁻¹ (RR) and 1120 g a.i. ha⁻¹ (DRR). Profenofos and triazophos were applied at the rate of 500 g a.i. ha⁻¹ (RR) and 1000 g a.i. ha⁻¹ (DRR). Untreated plots were sprayed with water only each treatment was replicate thrice. After the second spray, fruit samples (2 kg) from each replication were collected randomly at 0 (2 hours after spray), 1, 3, 5, 7 and 10 days intervals and transported to pesticide residue laboratory for pesticide residue analysis.

The brinjal fruit sample (2 kg) was homogenized in low speed high volume homogenizer (Robot coupe: Blixer® 6 V. V, France) and analysed as per the modified QuEChERS method (Mishra, 2011). Homogenized 15 g sample was taken in 50 ml polypropylene centrifuge tube containing 30 ml acetonitrile. Sample is homogenized at 15000 rpm for 3 minutes using low volume high speed homogenizer (Heidolph silent crusher: Heidolph, Germany). Then 10 g sodium chloride was added, shaken tube at 50 rpm for 3 minutes with Rotospin (Rotospin mixer: Tarson Products Pvt. Ltd) and centrifuged at 2500 rpm for 3 minutes in eppendorf centrifuge (Centrifuge: Eppendorf India Ltd.) to separate the organic layer. The top organic layer of about 15 ml was taken into the 50 ml polypropylene centrifuge tube containing 10 g anhydrous sodium sulphate to remove the moisture content. After removing moisture content, 6 ml of extract was taken in to 15 ml polypropylene centrifuge tube, containing 0.15 g PSA (Primary Secondary Amines) sorbent and 0.9 g anhydrous magnesium sulphate and 0.05 g Graphitized Carbon Black. The sample tube was capped, shaken for 3 minutes at 50 rpm in Rotospin mixer and then centrifuged for 10 minutes at 2500 rpm. The extract of about 4 ml was transferred into test tubes and evaporated to dryness in the TurboVap® LV concentration Work station (Caliper Life Sciences) at 45°C in the presence of nitrogen current. Reconstituted the volume with 2 ml n-hexane and injected 1 µl into gas chromatograph (GC) equipped with flame photometric detector (FPD).

After second spraying of the brinjal crop, soil samples (1 kg) were collected at 0, 3, 5 and 10 days interval and analysed by QuEChERS technique (Asensio-Ramos *et al.*, 2010). A representative 10 g sieved ground dry soil sample was taken in a 50 ml polypropylene centrifuge tube containing 20 ml acetonitrile and shaken for 1 minute at 50 rpm using a Rotospin shaker. Tube was centrifuged at 3300 rpm for 3 minutes after adding 4 g magnesium sulphate and 1 g sodium chloride. After centrifugation, a 10 ml of supernatant was taken in another 15 ml centrifuge tube containing 1.5g of magnesium sulphate and 0.250 g of PSA. Tube was shaken for 3 minutes at 50 rpm in Rotospin. After shaking, the tube was sonicated for 3 minutes in sonicator (PCI Analytical Pvt. Ltd.) and centrifuged for 10 minute at 4400 rpm. Aliquot of 4 ml was withdraw in a turbo tube and evaporated to dryness in presence of nitrogen current at 45°C in the TurboVap® LV concentration Work station. The residues were dissolved in 2 ml of n-hexane and injected 1 µl into gas chromatograph.

The cleaned extract was analyzed on Shimadzu GC 2010 equipped with Capillary glass column, Rxi®-5ms (30 mt, 0.25 mm ID, 0.25 µm film thickness) coupled with flame photometric detector (FPD). The oven operating parameters

in a multiramp system were: For acephate- initial temperature 80°C held for 1 minute then increased to 150°C @ 5°C and kept for 2 minutes. Temperature again increased to 250°C @ 5°C and held for 2 minutes. For profenofos and triazophos- initial temperature 80°C held for 3 minutes and then increased to 250°C @ 20°C and held for 20 minutes. Under these operation parameters, retention time of acephate, profenofos and triazophos was 17.124, 16.252 and 18.117 minutes.

The analytical method employed to estimate acephate, profenofos and triazophos residues was validated by spiking the control fruit and soil samples at five different concentrations viz., 0.05, 0.10, 0.25, 0.50 and 1.00 mg kg⁻¹ fortification levels each spiking level was replicated thrice. The limit of determination (LOD) for acephate, profenofos and triazophos was 0.05 mg kg⁻¹. The residue data were subjected to statistical analysis (Hoskins, 1961).

Analytical grade reagents viz. acetone, acetonitrile, n-hexane, magnesium sulfate, sodium chloride, sodium sulphate were obtained from M/S Merck Specialties Private Limited, Mumbai, India. Graphitized Carbon Black (GCB) was obtained from Crescent Scientific Pvt. Ltd. Mumbai. Primary secondary amine (PSA) was procured from M/S Agilent Technologies Pvt Ltd, Worli, Mumbai. Certified reference material of acephate, profenofos and triazophos were procured from Dr Ehrestorfer lab, Germany.

RESULTS AND DISCUSSION

Data presented in Table 1 depicts reliability of analytical method tested by spiking of untreated brinjal fruits and soil samples at different concentrations. Recovery of acephate was between 88.66-106.00 per cent in fruits and 82.00-92.33 per cent in soil fortified samples. Recovery of profenofos was between 84.00-97.30 per cent in fruits and 82.00-96.33 per cent in soil fortified samples. Recovery of triazophos was between 86.93-103.38 per cent in fruits and 81.33-93.66 per cent in soil fortified samples. The results are in agreement with investigations of Trevizan *et al.* (2005) who observed 83-117 and 73-116 per cent recovery of acephate in okra fruits and soil respectively. The recovery of acephate from mango orchard soil was in range of 89.75-91.44 per cent (Mohapatra *et al.*, 2011). The recoveries of profenofos were 80.43-93.29 per cent from tomato fruits and 77.71-95.46 per cent from tomato cropped soil at 0.005-0.5 µg g⁻¹ fortification levels (Gupta *et al.*, 2011). Singh *et al.* (2015) observed recovery of triazophos between 85.92-92.87 per cent in capsicum fruits. Lingyan *et al.* (2009) obtained 80.0–111.1 per cent recovery of triazophos from soil at 10.0-50.0 ng g⁻¹ spiking levels.

The average initial deposits of acephate on brinjal were found to be 0.683 and 1.080 mg kg⁻¹ at 560 and 1120 g a.i. ha⁻¹ respectively. The initial deposits were dissipated to 0.110 and 0.206 mg kg⁻¹ after 5 days at single and double dosages, respectively, thereby showing losses of about 83.90 and 80.84 per cent. Residues of acephate dissipated below the determination limit of 0.05 mg kg⁻¹ after 7 days at the recommended dose, but a 0.053 mg kg⁻¹ residue was present after 7 days on the sample with double the recommended dose (Table 2). The present findings are in agreement with those of Mohapatra *et al.* (2011) who reported an initial deposit

Table 1 : Recovery of acephate, profenofos and triazophos from brinjal fruits and soil samples

	Fortification level, (mg kg ⁻¹)	Fruits Mean Recovery (%)	Relative standard deviation (%RSD)	Soil Mean Recovery (%)	Relative standard deviation (%RSD)
Acephate	0.05	88.66	0.003	82	0.001
	0.1	96.66	0.006	85.33	0.006
	0.25	91.33	0.017	84.66	0.007
	0.5	84.66	0.032	88	0.02
	1	106	0.026	92.33	0.045
Profenofos	0.05	96	0.007	94	0.002
	0.1	92.66	0.002	85.33	0.005
	0.25	96	0.026	82	0.005
	0.5	84	0.01	82.66	0.015
	1	97.3	0.017	96.33	0.015
Triazophos	0.05	90.66	0.002	81.33	0.005
	0.1	89.66	0.001	88.33	0.007
	0.25	86.93	0.002	86.66	0.015
	0.5	94.8	0.009	86.66	0.015
	1	103.38	0.059	93.66	0.023

Table 2 : Persistence of acephate in/on brinjal fruit at the application rate of 560 and 1120 g a.i. ha⁻¹

Interval (Days)	Dose 560 g a.i. ha ⁻¹ Mean residue \pm SD	Dissipation (%)	Dose 1120 g a.i. ha ⁻¹ Mean residue \pm SD	Dissipation (%)
0	0.683 \pm 0.005		1.080 \pm 0.010	
1	0.546 \pm 0.015	20	0.813 \pm 0.015	24.69
3	0.286 \pm 0.049	58.04	0.453 \pm 0.020	58.02
5	0.110 \pm 0.010	83.9	0.206 \pm 0.020	80.86
7	0.050 \pm 0.000	92.68	0.090 \pm 0.010	91.66
10	BDL		0.053 \pm 0.005	95.06
15	BDL		BDL	
Control	ND		ND	
Regression equation	Y = -0.113-0.166X		Y = 0.0309-0.138X	
Correlation coefficient (r)	-0.995		-0.992	
RL ₅₀ (Days)	1.8		2.2	

Table 3: Persistence of profenofos in/on brinjal fruit at the application rate of 500 and 1000 g a.i. ha⁻¹

Interval (Days)	Dose 500 g a.i. ha ⁻¹ Mean residue \pm SD	Dissipation (%)	Dose 1000 g a.i. ha ⁻¹ Mean residue \pm SD	Dissipation (%)
0	1.966 \pm 0.015		2.460 \pm 0.010	
1	0.950 \pm 0.000	51.69	1.063 \pm 0.005	56.77
3	0.416 \pm 0.005	78.81	0.696 \pm 0.011	71.68
5	0.190 \pm 0.010	90.33	0.363 \pm 0.006	85.23
7	0.070 \pm 0.000	96.44	0.087 \pm 0.006	96.47
10	BDL		0.070 \pm 0.000	97.15
15	BDL		BDL	
Control	ND		ND	
Regression equation	Y = 0.237-0.198X		Y = 0.283-0.157X	
Correlation coefficient (r)	-0.996		-0.971	
RL ₅₀ (Days)	1.5		1.9	

of 0.684 mg kg⁻¹ and 1.109 mg kg⁻¹ following application of acephate @ 0.75 and 1.5 kg a.i. ha⁻¹ respectively. Half-life of acephate was observed to be 1.8 and 2.2 days, respectively, when applied at 560 and 1120g a.i. ha⁻¹. According to Chuanjiang *et al.* (2010) and Kaur *et al.* (2015) the half life of acephate was 1.4 days in pakchoi and 1.55-1.52 days in brinjal crops. Maximum residue limit (MRL) of acephate on brinjal had is not available hence, a safe waiting period of chlorpyrifos on brinjal was calculated at the limit of determination of 0.05 mg kg⁻¹ and worked out to be at RR and DRR was 7.9 and 9.4 days, respectively. Reddy and Rao (2005) suggested 5.13 days waiting period on treated grape berries.

A perusal of the Table 2 revealed that profenofos initial deposits of 1.966 and 2.460 mg kg⁻¹ at RR and DRR applications, respectively. These deposits dissipated to 0.190 and 0.363 mg kg⁻¹ after 5 days at single and double dosages, respectively, thereby showing losses of about 90.33 and 85.23 per cent. These initial residues were dissipated to 96.44 and 97.15 per cent at the recommended and double the recommended dosages, respectively, after 7 and 10 days of the last application of the pesticide. Sahoo *et al.* (2004) observed an initial deposit of 1.37 mg kg⁻¹ and 2.52 mg kg⁻¹ following application of profenofos @ 0.500 and 1.000 kg a.i. ha⁻¹ respectively. The half-life of profenofos deposits on brinjal was 1.5 and 1.9

Table 4 : Persistence of triazophos in/on brinjal fruit at the application rate of 500 and 1000 g a.i. ha⁻¹

Interval (Days)	Dose 500 g a.i. ha ⁻¹		Dose 1000 g a.i. ha ⁻¹	
	Mean residue ± SD	Dissipation (%)	Mean residue ± SD	Dissipation (%)
0	1.100 ± 0.010		2.233 ± 0.006	
1	0.783 ± 0.028	28.78	1.050 ± 0.020	52.98
3	0.386 ± 0.006	64.84	0.640 ± 0.010	71.34
5	0.183 ± 0.006	83.33	0.240 ± 0.000	89.25
7	0.053 ± 0.006	95.15	0.086 ± 0.006	96.11
10	BDL		0.070 ± 0.000	96.86
15	BDL		BDL	
Control	ND		ND	
Regression equation	Y = 0.086-0.182X		Y = 0.231-0.155X	
Correlation coefficient (r)	-0.991		-0.973	
RL ₅₀ (Days)	1.6		1.9	

Table 5 : Residues of acephate, profenofos, and triazophos in field soil following application on brinjal crop

Insecticide	Rate (g a.i. ha ⁻¹)	Mean Residue (mg kg ⁻¹) ± SD				
		0 Day	3 Day	5 Day	10 Day	15 Day
Acephate	560	0.270 ± 0.020	0.067 ± 0.001	BDL		
	1120	0.473 ± 0.005	0.133 ± 0.015	0.060 ± 0.017	BDL	
Profenofos	500	0.950 ± 0.030	0.336 ± 0.015	0.173 ± 0.020	BDL	
	1000	1.423 ± 0.015	0.623 ± 0.020	0.463 ± 0.023	0.213 ± 0.011	BDL
Triazophos	500	0.493 ± 0.025	0.166 ± 0.015	0.096 ± 0.011	BDL	
	1000	1.186 ± 0.025	0.486 ± 0.015	0.336 ± 0.020	0.163 ± 0.023	BDL

days at recommended and double recommended doses. Banshtu *et al.* (2015) reported the half-life values of profenofos deposits on tomato as 2.02 days at lower and 2.22 days at higher doses. Based on the persistence study and limit of determination of 0.05 mg kg⁻¹ the safe pre-harvest interval recommended for consumption of brinjal fruits from treatment at recommended and double dose were 6.3 and 7.9 days, respectively. Nigam *et al.* (2009) suggested safe waiting period of 6.22 and 7.00 days for profenofos on brinjal fruits at recommended and double the recommended doses, respectively.

It was observed that application of triazophos on brinjal crop leads to 1.100 and 2.233 mg kg⁻¹ initial deposits on brinjal fruits at RR and DRR, respectively. The initial deposits dissipated to 0.183 and 0.240 mg kg⁻¹ after 5 days at single and double dosages, respectively, thereby showing losses of about 83.33 and 89.25 per cent. Brinjal being a smooth surface crop had lower initial deposit in comparison to the findings of Singh (2013) who reported initial deposit to the tune of 1.870 and 3.076 mg kg⁻¹ at the same application rate on capsicum. The half-life of triazophos deposits on brinjal fruits were 1.6 and 1.9 days following the application of triazophos at the rate of 500 and 1000g a.i. ha⁻¹, respectively. Shashi *et al.* (2015) and Cherukuri *et al.* (2015) observed 1.14 days RL₅₀ value of triazophos deposits on brinjal and tomato crop. A safe waiting period worked out at the limit of determination of 0.05 mg kg⁻¹ was at 7.1 and 8.0 days for RR and DRR doses, respectively. Singh *et al.* (2015) who suggested waiting period of 7 days from consumer's safety point of view.

The initial deposit of acephate residues in soil was 0.270 and 0.473 mg kg⁻¹ at the recommended and double the recommended dosages, respectively. Residues dissipated with time and no residues were detected in samples collected after 5th and 10th day at the recommended and double the recommended dosages, respectively. Chai *et al.* (2009) who

showed that acephate dissipated completely from mustard cropped soil at 7-14 days after last spraying. The initial deposit of profenofos observed to be 0.950 and 1.423 mg kg⁻¹ and triazophos 0.493 and 1.186 mg kg⁻¹ at RR and DRR dose, respectively. Residue of profenofos and triazophos were further reduced below the determinable limit on day 10th and 15th (Table 5). Gupta *et al.* (2011) and Mukherjee *et al.* (2012) detected no residues of profenofos in soil samples collected after 7th day from soils cropped with tomato and eggplant. Singh (2013) reported that triazophos disappeared in soil of capsicum by 10th and 15th days at the single and double doses, respectively.

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5. **The best oration award** : It is awarded to the scholar who delivered invited speech.
6. **The recognition award** : It is awarded to those senior scholars who have contributed to the subject through their continued research .
7. **The environmental awareness award** : It is awarded to those who, apart from their research contribution, have done commendable extension work for environmental betterment.

The number of recipients of award in each category will vary depending upon the recommendation of the panel of judges and the executive committee. The association has the provision to institute awards in the name of persons for whom a with desired sum is donated in consultation with the executive body.

PUBLICATION OF THE ASSOCIATION

In order to provide a platform to a vast group of researchers to express their views and finding of research as well as to promote the attitude of quality research among the scholars of younger generation the association publishes an international quarterly journal – **THE BIOSCAN (ISSN:0973-7049)**. For the benefit of the potential contributors **instructions to authors** is given separately in this journal. However, the details regarding the journal and also the association can be seen on our website www.thebioscan.in.

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