Indian Journal of Agricultural Sciences **88** (2): 240–4, February 2018/Article

Residue behaviour of profenofos and triazophos in okra (*Abelmoschus esculentus*) and their decontamination using culinary processes

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Received: 02 December 2016 ; Accepted: 17 October 2017

ABSTRACT

The persistence pattern and risk assessment of profenofos and triazophos in okra (*Abelmoschus esculentus* (L.) Moench) and cropped soil were studied in the present study. The insecticides were applied twice at 10 days interval @ 500 and 1000 g a.i./ha. Residues were quantified at 0, 1, 3, 5, 7, 10, 15 and 20 days of second application using GC equipped with Flame Photometric Detector (FPD). The average initial deposits of triazophos (1.838 mg/kg) in okra fruits were higher than profenofos (1.418 mg/kg) at the recommended application rates. Both the insecticides followed a first order kinetics with half-lives of 1.6 and 1.4 days, respectively on okra fruits. The initial deposits of 0.483 and 0.500 mg/kg, for respective insecticides, disappeared to BDL on 5th and 7th day in okra cropped soil. Microwave oven cooking proved more promising than other household processing in dislodging the residues of test insecticides from okra fruits. On the basis of this study, waiting periods of 7.6 and 7.4 days were suggested for consumption of okra sprayed with profenofos and triazophos, respectively.

Key words: Household processes, Okra, Organophosphates, Persistence, Risk assessment

Okra [*Abelmoschus esculentus* (L.) Moench] is one of the important vegetable crops grown throughout the tropical and subtropical parts of the world. Okra is valued for its delicious and nutritious edible green pods. India is the leader in production of okra in the world with 533 thousand hectare area and 6346 thousand tonne production. In Himachal Pradesh, it is grown on an area of 2.76 thousand hectare with 34.03 thousand tonne production (Anonymous 2016). Okra has a vast potential as one of the foreign exchange earning crop and accounts for about 60% of the India's export of fresh vegetables excluding potato, onion, garlic etc. (Dhall *et al.* 2014). Organophospates such as profenofos and triazophos are extensively used insecticides in okra for the control of insect-pests without having any approval or recommendations in the country, and their residues could pose a great health problem to the consumers. We found 2.7% of the vegetable samples collected from various Indian markets exceeded the proposed Maximum Residue Limits (MRL) of various pesticides. Also, the vegetable samples collected from farm-gate and organic outlets exceeded MRLs by 3.7 and 2.0%, respectively (Anonymous 2015).

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Trading food products requires adherence to safety standards that include pesticide MRLs. MRLs ensure that imported and exported food is safe to eat. Therefore, establishing MRLs for crops is very important especially for developing countries as the lack of residue limits for exported products could create barriers to trade (Kunkel 2012, Ferro *et al.* 2015). Therefore, to generate the information on persistence and residues of profenofos and triazophos in okra, the present study was conducted as per Good Agricultural Practices (GAP). The information generated would help fixing up MRL and PHI for label claim. Insecticides applied on the crop ultimately get their way into the soil and affect the soil micro flora and fauna, render poor soil fertility and finally affect crop yield. So, persistence of these insecticides in soil was also studied. Besides this, the impact of some household culinary methods like washing and cooking on reduction of insecticides residues from okra fruits was also studied.

MATERIALS AND METHODS

The field trials were conducted on okra (var. Punjab-8) in the Experimental Farm, Department of Entomology, Dr YSP University of Horticulture and Forestry, Navai, Solan following good agricultural practices. The experiments were laid out in randomized block design (RBD) with three replications along with control. The crop was raised as per the package and practices of the University.

Profenofos (Profex 50 EC) and Triazophos (Rider 40 EC) were sprayed twice at 10 day interval @ 500 and 1000

g a.i./ha on the crop using a Knapsack sprayer fitted with hollow cone nozzle. The lower concentration was sprayed first, followed by higher concentration.

Fruit samples (2 kg) were collected at each interval (0, 1, 3, 5, 7, 10, 15 and 20 days), after the second spray. Each sample was homogenized in a blender at a high speed to get the fine homogenate without any large particles or segregated material. Fruit samples were analyzed in the laboratory as per the QuEChERS (quick, easy, cheap, effective, rugged and safe) technique (Asensio-Ramos *et al.* 2010). A representative of 15 g homogeneous sub-sample was taken up with 20 ml acetonitrile in 50 ml centrifuge tube and homogenized at 15000 rpm for 2 min by using low volume high speed homogenizer. The homogenizer was washed with 10 ml acetonitrile and pooled in 20 ml fraction. Anhydrous sodium chloride (10 g) was added into the tube, shaken at 50 rpm for 5 min with Rotospin mixer and then centrifuged at 2500 rpm for 3 min. Upper layer fraction of 15 ml was transferred to another 50 ml centrifuge tube containing 10 g anhydrous sodium sulphate and was shaken for 5 min at 50 rpm.

Anhydrous magnesium sulphate (900 mg), PSA (150 mg) and GCB (50 mg) were taken in 15 ml centrifuge tube. The tube was capped and shaken for one minute on Spinix test tube shaker. Six ml fractions from 15 ml extract was added into the centrifuge tube, rotated for one minute at 50 rpm in Rotospin mixer and centrifuged at 2500 rpm for 10 minutes. The 4 ml fraction was transferred to the 30 ml turbo tube and evaporated in turbo- evaporator to dryness (at 45° C) in presence of nitrogen current. The residues were dissolved in 2 ml n-hexane for gas chromatographic (GC) analysis.

A gas chromatograph equipped with Flame Photometric Detector (FPD) and capillary glass column, DB-5 (30 meter, 0.25 mm ID, 0.25 µm film thickness) was used for estimation of residues. The oven temperature was programmed at 80°C for 3 min, raised to 250°C @ 20°C per min and held for 20 min. The temperature of injection port and detector was kept at 250°C and 280°C, respectively. Gas flow: 80.0 ml/min (hydrogen), 120.0 ml/min (zero air) and 0.9 ml/min (nitrogen). Under these set of conditions, the retention time of profenofos and triazophos was 17.07 and 19.27 minutes, respectively.

Representative fruit samples of single dose (500 g a.i./ha) from each treatment collected at 1, 3 and 5 days after spray were used to undertake the decontamination studies. The culinary processes, viz. washing with tap water (for 2 minutes and then dried in shade), saline water washing (fruits were dipped in water containing 2% NaCl solution) and after 5 min, the fruits were gently rubbed by hands in salt solution and dried in shade), lukewarm water washing (fruit samples were dipped in lukewarm water (35-40°C) for 5 min, gently rubbed by hands and dried in shade), open pan cooking (fruit samples were chopped into small pieces, cooked in an open pan till softness) and microwave cooking (samples were cut into small pieces and cooked at 1400 W power output in microwave for 5 min) were used. The fruit samples processed with the above mentioned culinary methods were analysed for residue estimation as per already described procedure of QuEChERS technique. The residue data were subjected to statistical analysis according to Hoskins (1961) to compute the residual half-life (RL_{50}) and safe pre-harvest interval (PHI).

RESULTS AND DISCUSSION

Recovery studies

The method of analysis of profenofos and triazophos was validated in the laboratory by carrying out the recovery experiments at fortification levels of 0.05, 0.10, 0.25, 0.50 and 1.0 mg/kg. Based on these recovery studies, the limits of determination (LOD) was 0.05 mg/kg for profenofos and triazophos. The recoveries for profenofos varied from 93.00-113.00 and 91.00-101.00%, respectively from fortified fruits and soil. For triazophos, these were 92.35-112.00 and 88.20-116.50%, respectively in fruits and soil (Table 1). The method of analysis was found to be satisfactory as the recoveries of the test insecticides were above 80%. The half life (RL_{50}) and safe waiting period were worked out as per Hoskins formulae (1961). Since MRL of the test insecticides in okra were not available in the literature (Codex Elemetarious/PFA). Hence, the waiting periods of these insecticides in okra were calculated by considering LOQ as 0.05 mg/kg as MRL.

Persistence of profenofos and triazophos in okra fruits

The data for residue of profenofos and triazophos in okra are presented in Table 2. The initial deposits (2 h after application) of profenofos on okra fruits were 1.418 and 2.801 mg/kg at recommended and double the recommended dosages, respectively. The residues dissipated with time and by 10 days; residues were below detectable limit at the recommended dose of application, while in the double the recommended dose of application, the residues dissipated below detectable limits within 15 days recording a loss of 95-97 percent. Dissipation of profenofos followed first-order kinetics and registered half-life values of 1.6 and 1.9 days with 7.6 and 11.2 days safe waiting periods at respective dosages. These results are in accordance with Radwan *et al.* (2005) who observed half life values of profenofos in hot pepper, sweet pepper and aubergine as 1.84, 1.74

Table 1 Recovery of profenofos and triazophos from fortified okra fruits and cropped soil

Fortification levels (mg/kg)	Recovery (%)			
	Profenofos		Triazophos	
	Fruits	Soil	Fruits	Soil
0.05	96.00	94.00	110.00	106.00
0.10	93.00	100.00	112.00	110.00
0.25	106.40	91.20	97.20	98.80
0.50	113.40	101.40	94.60	88.20
1.00	111.40	97.70	92.35	116.50

Table 2 Dissipation of profenofos and triazophos residues from okra fruits

Interval (Days)	Average residue (mg/kg) ± SD			
	Profenofos		Triazophos	
	500 g a.i./ha	1000 g a.i./ha	500 g a.i./ha	1000 g a.i./ha
0	1.418 ± 0.071	2.801 ± 0.243	1.838 ± 0.031	3.092 ± 0.022
1	0.819 ± 0.016 (42.24)	1.119 ± 0.094 (60.08)	0.942 ± 0.025 (48.80)	1.268 ± 0.018 (58.99)
3	0.269 ± 0.005 (81.02)	0.535 ± 0.053 (80.93)	0.360 ± 0.020 (80.41)	0.684 ± 0.071 (77.87)
5	0.136 ± 0.007 (90.40)	0.244 ± 0.002 (91.28)	0.162 ± 0.010 (91.07)	0.263 ± 0.014 (91.49)
7	0.064 ± 0.007 (95.48)	0.110 ± 0.000 (96.07)	0.054 ± 0.002 (97.06)	0.085 ± 0.005 (97.25)
10	BDL	0.074 ± 0.001 (97.35)	BDL	0.056 ± 0.004 (98.18)
15	BDL	BDL	BDL	BDL
Control	ND	ND	ND	ND
r	-0.994	-0.973	-0.997	-0.980
RL_{50} (days)	1.6	1.9	1.4	1.7
Waiting period (days)	7.6	11.2	7.4	10.2
Regression equation	Y= 0. 098-0.191X	Y= 0.261-0.155X	Y= 0.086-0.182X	Y= 0.352-0.176X

BDL - below detectable level (0.05 mg/kg), ND - not detected. Figures in parenthesis indicate % dissipation

and 1.96 days, respectively. Shah *et al.* (1999) and Nath *et al.* (2005) also reported half-life value for profenofos initial deposits as 1.35 days in okra fruits. Similar results were obtained by Mukherjee *et al.* (2012), who calculated half-life value of profenofos in brinjal fruits as 0.91–1.86 days. Nigam *et al.* (2009) suggested waiting period of 6.22 and 7.00 days for recommended and double the recommended doses of profenofos respectively, on brinjal, which support the present investigation. Similarly, waiting periods of 7.8-11.1 days on cardamom (Renuka *et al.* 2006), 5 days on okra (Parmar *et al.* 2015), 15 days on brinjal (Patel *et al.* 2015) and 10 days on capsicum (Joshi *et al.* 2015) for profenofos were recorded by different workers.

In case of triazophos, initial deposits of 1.838 and 3.092 mg/kg at recommended and double the recommended dosages, respectively were recorded on okra fruits (Table 2). Similar to profenofos, triazophos also dissipated in 10-15 days below detectable limit with 97-98 per cent loss of residue. A residue half-life of 1.4 and 1.7 days was calculated for triazophos on okra fruits at respective application rates. Based on these observations, safe waiting period of 7.4 and 10.2 days at respective dosages are suggested for triazophos on okra fruits. These findings are supported by Raj *et al.* (1999), who showed 1.0 day half life of triazophos in okra fruits and 1.0-2.0 days in brinjal fruits. Patel *et al.* (2015) observed 2.85 days half-life of triazophos on brinjal, which is also in accordance with the present investigations. Similar results were obtained by Banerji *et al.* (2008), who calculated half-life value of triazophos in bitter gourd fruits as 0.75-1.55 days. These values are closer to those obtained by Kumar *et al.* (2000) and Parmar *et al.* (2015) who reported 6.33 and 5 days waiting period for triazophos treated chilli and okra fruits, respectively. The present studies also find support by Singh *et al.* (2015) who observed a half-life of 2.31 days and waiting period of 7 days on capsicum sprayed with triazophos at the recommended dosages.

Dissipation of profenofos and triazophos from okra cropped soil

Persistence, degradation, leaching and dispersion of pesticides in the soil environment depend not only on the properties of pesticides and soil but also on the prevailing climatic conditions (Vig *et al.* 2001). The main processes which potentially affect the ultimate fate of pesticides in soil are adsorption/desorption processes, transformation processes (biological and chemical degradation) and transportation through soil, atmosphere, surface water, or ground water (Kumari *et al.* 2008). It is evident from this

Table 3 Dissipation of profenofos and triazophos residues from okra cropped soil

Interval (Days)	Average residue (mg/kg) ± SD			
	Profenofos		Triazophos	
	500 g a.i./ha	1000 g a.i. ha^{-1}	500 g a.i./ha	1000 g a.i./ha
0	0.483 ± 0.005	1.003 ± 0.011	0.500 ± 0.020	1.203 ± 0.015
3	0.090 ± 0.001 (81.25)	0.460 ± 0.010 (54.00)	0.200 ± 0.010 (54.00)	0.533 ± 0.011 (55.83)
5	BDL	0.096 ± 0.001 (90.40)	0.080 ± 0.001 (83.40)	0.220 ± 0.010 (81.71)
7	BDL	BDL	BDL	0.085 ± 0.001 (92.91)
10	BDL	BDL	BDL	BDL
Control	ND	ND	ND	ND
r	-0.928	-0.974	-0.969	0.979
RL_{50} (days)	2.4	3.9	3.0	3.7
Regression equation	Y= -0.065 - 0.108X	Y= 0.112 - 0.075X	Y= -0.377 - 0.099X	Y= -0.023 - 0.079X

BDL - below detectable level (0.05 $mgkg^{-1}$), ND - not detected Figures in parenthesis indicate % dissipation

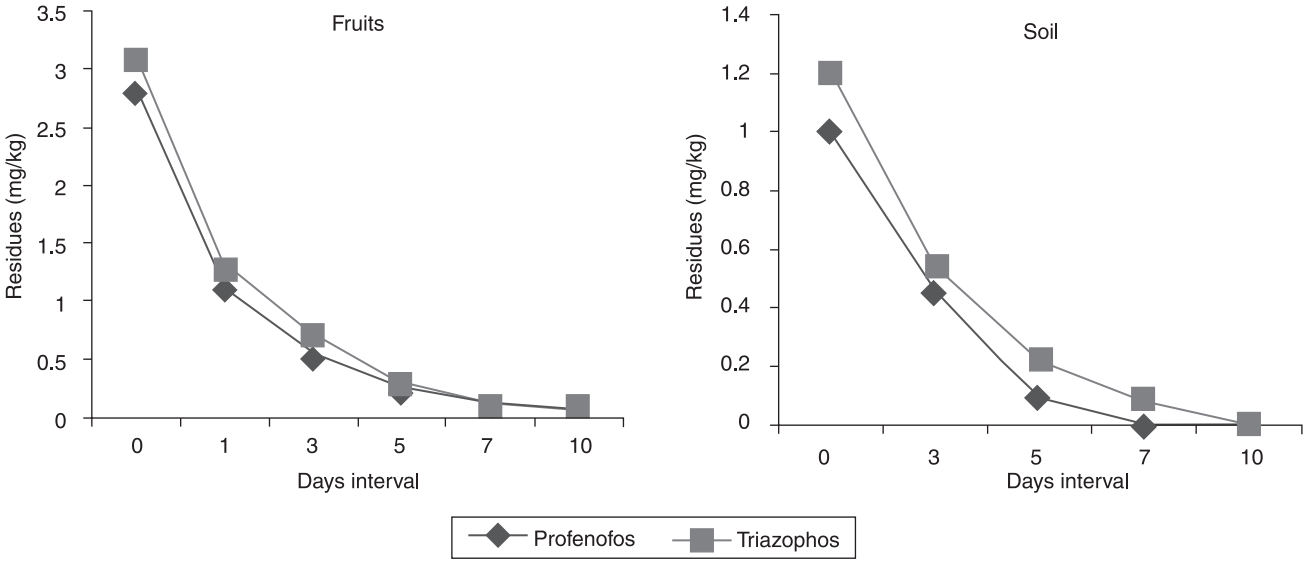

Fig 1 First order dissipation of insecticides in/on okra fruits and cropped soil at double doses

study that that profenofos and triazophos dissipated more than 80 per cent in 3 and 5 days, respectively at single dose (Table 3). It took 5 and 7 days to dissipate more than 90 percent at double dose. The residue half-life calculated was 2.4 and 3.0 days for triazophos and profenofos, respectively at recommended doses. These results are in accordance with Jiang *et al.* (2010) who reported 3.75 days half-life of profenofos in paddy soil. Results obtained by Bajeer *et al.* (2016) showed little deviation from present studies as they reported 9.1 days half-life of triazophos in various types of soils.

Decontamination studies

The effects of food processing on pesticide residue levels may be influenced by the physical location of the pesticide residue as well as the physico-chemical properties of the pesticide such as solubility, volatility, hydrolytic rate constants, water–octanol partition coefficient and thermal degradation (Keikotlhaile *et al.* 2010). According to Street (1969) loosely held residues of several pesticides are removed with reasonable efficiency by varied type of washing processes. In present investigations, effect of different methods of washing on fate of test insecticide residues revealed that lukewarm water washing reduced residues of profenofos (31.25-56.20%) from okra fruits more efficiently than washing with running tap water (22.71-40.60%) and with salt water (27.10-51.09%) (Fig. 1). Similarly, in case of triazophos lukewarm water resulted in higher relief (46.86-59.64%) than washing with simple running tap water (33.79-42.59%) and salt water (36.23-50.61%).

The loss of pesticide residue during heat processing may be due to evaporation, co-distillation and thermal degradation which vary with the chemical nature of the

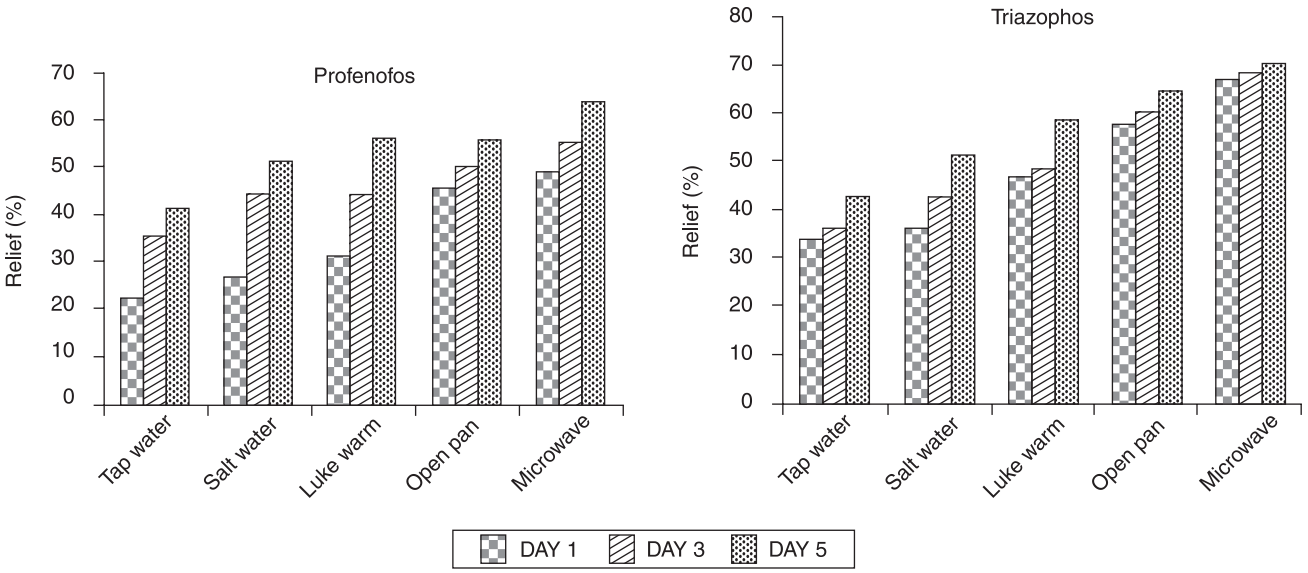

Fig 2 Effect of culinary processes on profenofos and triazophos residues in okra fruits

individual pesticide (Bajwa and Sandhu 2014). In present studies, open pan cooking of okra fruits provided relief up to 45.90-56.20 and 57.49-64.81 per cent from residues of profenofos and triazophos, which enhanced to 49.08-63.50 and 66.59-70.00 per cent in microwave cooking, respectively. Cooking methods proved more promising than different type of washings in dislodging the residues of test insecticides from okra fruits (Fig 2). In a similar study, Parmar *et al.* (2012) also reported a higher dislodging of profenofos and triazophos residues from okra due to cooking (46.62 to 95.10%) than washing with 2% brine solution (31.58 to 94.67%), washing with normal water (26.32 to 93.72%), dipping in normal water (9.02 to 90.80%) and rubbing with wet cloth (0.93 to 32.69%).

ACKNOWLEDGMENTS

We sincerely acknowledge the grants received from the Network Coordinator, AINP on Pesticide Residues, ICAR New Delhi.

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