Journal of Pharmacognosy and Phytochemistry
Available online at www.phytojournal.com

E-ISSN: 2278-4136
P-ISSN: 2349-8234
JPP 2019; 8(2): 787-789
Received: 18-01-2019
Accepted: 20-02-2019

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Dissipation and persistence of profenophos in/on tomato

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Abstract
Field trial was conducted to study the dissipation pattern of profenophos in/on tomato by following two foliar applications at recommended (500 g a.i/ha) and double the recommended dose (1000 g a.i/ha). The tomato crop was sprayed twice at 10 days interval at fruit initiation stage. Initial residues of profenophos in tomato fruits were recorded as 0.45 and 95 mg kg^{-1} on tomato fruits with the half-life of 2.56 and 2.56 days at recommended and double recommended dose, respectively. The residues of profenophos reached below quantification limit (BQL) after 7 and 10 days in both the doses. Considering this, Pre-Harvest Interval (PHI) of seven days can be suggested for profenophos for residue free tomato.

Keywords: Persistence, profenophos, tomato

Introduction
India is the second largest producer of tomato after China producing 18653 thousand MT over an area of 907 thousand ha. With a productivity of 20.6 MT ha^{-1} (Anon., 2014). In India, it is grown in a wide range of climatic conditions across states of Bihar, Karnataka, Uttar Pradesh, Orissa, Andhra Pradesh, Maharashtra, Madhya Pradesh and Assam. Tomato crop is damaged by various insect and non-insect pests from nursery stage to harvest.
Tomato fruit borer (*Helicoverpa armigera* Hubner) is a polyphagous pest and distributed widely in Indian subcontinent (Singh and Narang, 1990) [10]. The yield of tomato throughout the country is considerably lower because the most damage caused due to fruit borer, *Helicoverpa armigera* (Hubner) (Sahoo, *et al*., 2004) [7]. It is also reported to be 40 to 50 per cent in Bangalore (Khanderkhan *et al*., 1997) [6] and 32.52 per cent in Madhya Pradesh (Ganguly and Dubey, 1998) [3].
Farmers rely heavily on pesticides for the control of insect pests. A huge amount of pesticides are used on vegetables and their irrational and continual use has the reason resulted in the accumulation of pesticide residues in the primary agricultural products as well as soil. (Baig *et al*., 2009) [2]. Their residues are frequently reported in tomato fruits collected from farm gate and also market. Present investigation was conducted to generate the information on dissipation of profenophos in tomato by following good agricultural practices. The information would help in fixing the Maximum Residue Limit (MRL) and Post-Harvest Interval (PHI) for label claim.

Material And Methods
Field experiment
A supervised field experiment for residue studies was conducted during *Rabi* -2016 at the Instructional Farm, Post Graduate Institute, Mahatma Phule Krishi Vidyapeeth, Rahuri, Ahmednagar. Tomato crop was raised by following recommended package of practices. Two sprays were given at an interval of 10 days, initiating the first spray at fruit initiation stage. According to residue studies protocol prescribed by Central Insecticidal Board and Registration Committee (CIB & RC), two doses of profenophos (500 g a.i. ha^{-1} and 1000 g a.i. ha^{-1}) were evaluated for their residues.

Chemicals and Reagents
Certified Reference Material of profenophos of high purity (98.6%) was obtained from Sigma Aldrich and commercial insecticide was purchased from local market of Rahuri. The solvents of HPLC grade were ethyl acetate obtained from Avant or 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

Primary stock solution, intermediate standard and working standards were prepared by dissolving reference standard (Purity – 98.6% and Make- Sigma- Aldrich) in ethyl acetate.

Method validation

Prior to analysis of samples, linearity of 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 profenophos 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. The tomato 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 (LOQ), 0.25 (5×LOQ) and 0.5 (10×LOQ) µg g^{-1} in triplicate. The extraction and clean up were performed as described earlier. Per cent recovery was calculated by using following formula.

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

Sampling

The tomato 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 tomato samples were brought to the laboratory in polythene bags and processed immediately.

Extraction and clean up

Treated tomato fruits were extracted by QuEChERS method (Sharma, 2013) [9]. 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. Homogenised 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 mentioned in Table 1.

Results and Discussion

Linearity

The results of linearity study are presented in Table 2 and linearity line was drawn. The response of the instrument was linear over the range tested and R^2 value was 0.996 for Profenophos (Fig. 1). These results indicated that the GC-FPD analysis is a valid method for residue determination of the tested insecticides in tomato fruits.

Recovery

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) [8] and mentioned in Table 3.

Dissipation of profenophos

The results revealed that there was reduction in residue levels of profenophos in/on tomato with time (Table 4. and Fig. 2). No residues were recorded in any tomato samples collected from untreated plots. At recommended dose of 500 g a.i. ha^{-1}, mean initial residues of profenophos were 0.45 mg kg^{-1} at two hr after second spray. Initial residues of 0.45 µg g^{-1} further dissipated to 0.33, 0.21, 0.10 and 0.06 mg kg^{-1} at 1, 3, 5 and 7 days, respectively and reached BQL at 10 days.

At double the recommended dose (1000 g a.i. ha^{-1}), mean initial residues of 0.95 mg kg^{-1} dissipated to 0.63, 0.54, 0.35, 0.12 and 0.06 mg kg^{-1} at 1, 3, 5, 7 and 10 days, respectively after second spray. Residues of profenophos dissipated to with a half-life of 2.51 and 2.56 days at 500 and 1000 g a.i. ha^{-1}, respectively.

The above findings are in agreement with Gupta *et al.* (2011) [4] and Katroju *et al.* (2014) [5]. Gupta *et al.* (2011) [4] studied dissipation of cypermethrin, chlorpyriphos and profenophos in tomato fruits and reported that residues of profenophos dissipated with half-life of 2.2-5.4 days in tomato fruits which was in agreement with the present finding. Katroju *et al.* (2014) [5] determined dissipation pattern of profenophos in tomato. They reported mean initial residues of 1.16 mg g^{-1} after application of profenophos 50 EC @ 1000 g a.i ha^{-1}. Further, the residues dissipated within 7 days with a half-life of 4.81 days. As there is no MRL available for profenophos tomato, 0.05 µg g^{-1} may be taken as a default MRL. On the basis of this, Pre Harvest Interval (PHI) of seven days can be suggested for profenophos for harvesting tomato fruits free from residues.

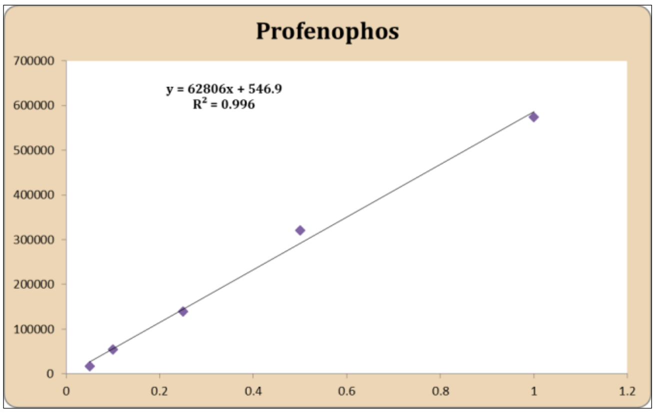

Fig 1: Linearity of profenophos standard

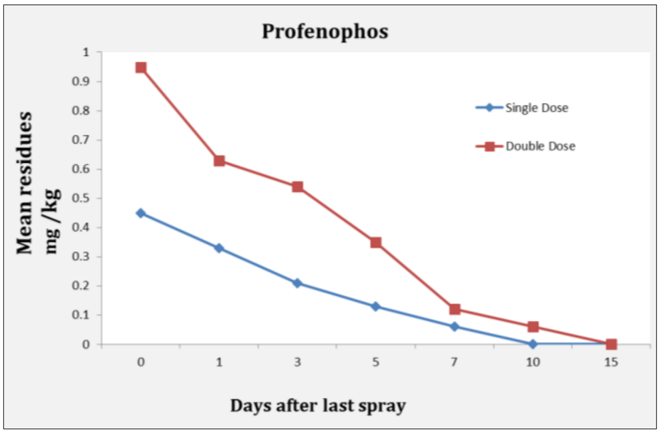

Fig 2: Dissipation pattern of profenophos residues in tomato

Table 1: Gas chromatographic parameters

Column	DB-1, 30m x 0.25 µm × 0.25 mm
Column Temperature	170 ^{0}C …… 3 min hold @ 6.5 ^{0}C/min 220 ^{0}C …… 2 min hold @ 10 ^{0}C/min 280 ^{0}C …… 6 min hold
Injector Temperature	250 ^{0}C
Column Temperature	170 ^{0}C
Detector Temperature	300 ^{0}C
Injection Volume	1 µl
Column flow	0.96 ml min^{-1}
Hydrogen Flow	90 ml min^{-1}
Air Flow	120 ml min^{-1}

Table 2: Linearity of profenophos standard

Compounds	Corresponding peak area				
	0.05 mg/kg	0.10 mg/kg	0.25 mg/kg	0.50 mg/kg	1.00 mg/kg
Triazophos	17196	54435	139191	321259	574329

Table 3: Recovery of profenophos in tomato fruits

Fortification Level (mg/kg)	Recovery (%)			
	R-I	R-II	R-III	Mean
0.05	87.32	94.57	87.78	84.41 (± 3.91)
0.25	80.82	95.17	95.46	95.18 (± 0.65)
0.50	85.09	95.81	100.25	94.50 (± 6.65)

Table 4: Residues of profenophos in tomato fruits

Interval between last application and sampling	Profenophos			
	Recommended dose		Double the recommended dose	
	Mean Residues (µg g^{-1})	Dissipation (%)	Mean Residues (µg g^{-1})	Dissipation (%)
0 day (2 hr.)	0.45 (±0.04)	-	0.95 (±0.13)	-
1 day	0.33 (±0.06)	26.66	0.63 (±0.05)	33.68
3 day	0.21 (±0.02)	53.33	0.54 (±0.10)	43.15
5 day	0.13 (±0.02)	71.11	0.35 (±0.08)	63.15
7 day	0.06 (±0.01)	86.66	0.12 (±0.02)	87.36
10 day	BQL	-	0.06 (±0.01)	93.68
15 day	BQL	-	BQL	-
$RL_{50(days)}$	2.51		2.56	

BQL - Below Quantification Level LOQ - 0.05 mg kg^{-1}

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