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The Dissipation Pattern, Residues of Profenophos In Tomato (*Lycopersicon esculentum* Mill.)

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ABSTRACT
Field experiment was carried out during kharif, 2012 to evaluate the dissipation pattern of most commonly used insecticide profenophos 50 EC @ 1000 g a.i. ha^{-1} with two sprays of insecticide, first given after fruit initiation and the second spray 10 days later and collecting the fruits at 0, 1, 3, 5, 7, 10, 15, 20 days after last spray, and analysed for residues using the validated QuEChERS method. The initial deposits of 1.16 mg kg^{-1} profenophos detected on tomato sprayed with profenophos @ 1000 g a.i. ha^{-1} were dissipated by 42.24% (0.67 mg kg^{-1}) by 1st day, 56% (0.51 mg kg^{-1}) by 3rd day, 73.25% (0.31 mg kg^{-1}) by 5th day and below determination level (0.05 mg kg^{-1}) by 7th day. A safe waiting period of 1 day is recommended considering Maximum Residues Limits (10 mg kg^{-1}) of Codex Alimentarius Commission (CAC).
Key words: *Tomato, Dissipation pattern, Profenophos*

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INTRODUCTION

Tomato (*Lycopersicon esculentum* Mill.) is one of the most important and remunerative vegetable crop grown in tropical and subtropical regions of the world for fresh market and processing, constituting an important part of our human diet. The consumption of tomato exceeds all vegetables and is next to Potato. In India, it is cultivated in an area of 865 thousand ha with an average annual production of 16826 thousand tonne and productivity of 19.5 t ha^{-1}.Andhra Pradesh ranks first in area (296.3 Thousand ha) and production (5926.2 thousand tones), while Karnataka ranks first in productivity with 34.3 t ha^{-1} [1]. Like other vegetables, it is more prone to insect pests and diseases mainly due to the tenderness and softness as compared to other crops resulting in low yield. It is devastated by an array of pests like jassids, aphids, tobacco caterpillar, flea beetles, spider mites, and fruit borer. Of which the fruit borer is of economic importance.

To control the fruit borer, different pesticides are being used in large quantities by farmers except in few cases where the crop is grown as per Good Agricultural Practices (GAP) for export purposes.

The food habits are also changed, as tomato is being consumed as salad, and hence food safety issues are very important. Hence, GAP to be recommended so as to reduce the pesticide load in food and environment.Considering the economic importance of the fruit, studies conducted to evaluate the dissipation pattern of profenophos on tomato so as to recommend the safe waiting periods based on the Maximum Residue Limits (MRLs).

MATERIALS AND METHODS

Field experiment was carried out to evaluate the dissipation pattern of selective insecticide against fruit borer (*Helicopverpa armigera(Hub.*)) on cabbage during *kharif* 2012 at Student's Farm, College of Agriculture, Rajendranagar, Hyderabad utilizing 8 treatments including untreated control replicated thrice. The first spray was given after fruit initiation and the second spray 10 days later and the further chemical dissipation studies were conducted.

Pesticide Residue Analysis Method

Preparation of working standards of profenophos

Certified Reference Materials (CRMs) of profenophos purchased from Dr. Erhenstorfer, Germany. Primary, intermediary and working standards were prepared from the CRMs using acetone and hexane as solvents. Profenophos working standards in the range of 0.01 ppm to 0.5 ppm were prepared in 10 ml calibrated graduated volumetric flask using distilled n-hexane as solvent. All the standards were stored in deep freezer maintained at -40^{0}C.

Limit of Detection and Linearity test

The working standards of profenophos were injected in Gas Chromatograph VARIAN GC 3800 with Electron Capture Detector (ECD) and Thermionic Specific Detector (TSD) for estimating the lowest quantity of profenophos which can be detected with injector split ratio of 1:10 under standard operating parameters as given below. For confirmatory analysis, both profenophos was analysed on both ECD and TSD as this pesticides can be detected on both detectors simultaneously using "Universal Y splitter" at the detector end. One micro litre of each working standard was injected for the study. The GC operating parameters for profenophs detection and estimation are presented in Table 1.

Table 1. Details of GC operating parameters

Gas Chromatograph	Gas Chromatography-VARIAN GC 3800
Column	VF-1ms Capillary Column 30 m length, 0.25 mm Internal Diameter, 0.25 m film thickness; 1% methyl siloxane
Column Oven (^{0}C)	240 (Isothermal)
Detectors	Electron Capture Detector (ECD) Thermionic Specific Detector (TSD)
Detector Temperature (^{0}C)	280
Injector Temperature (^{0}C)	260
Injector Status	Front Injector Type 1177 Split / Splitless Split ratio: 1:10
Carrier Gas	Nitrogen, Iolar II, Purity 99.99%
Carrier Gas Flow (ml min^{-1})	1 ml/min
Make-up Flow (ml min^{-1})	35 ml/min
Retention time (min)	Profenophos 11.77 min
Total run time (min)	30 min

Under the GC operational parameters given in Table 1. the retention times of profenophos is 11.77 min . Each working standards of profenophos (0.01 ppm, 0.025 ppm, 0.05 ppm, 0.075 ppm, 0.10 ppm, 0.25 ppm and 0.50 ppm) were injected 6 times and the linearity lines were drawn. Based on the response of the detector (ECD), it is observed that the LOD (limit of detection) for profenophos is 0.01 ng, and the linearity is in the range of 0.01 ppm to 0.10 ppm, (Fig.1).

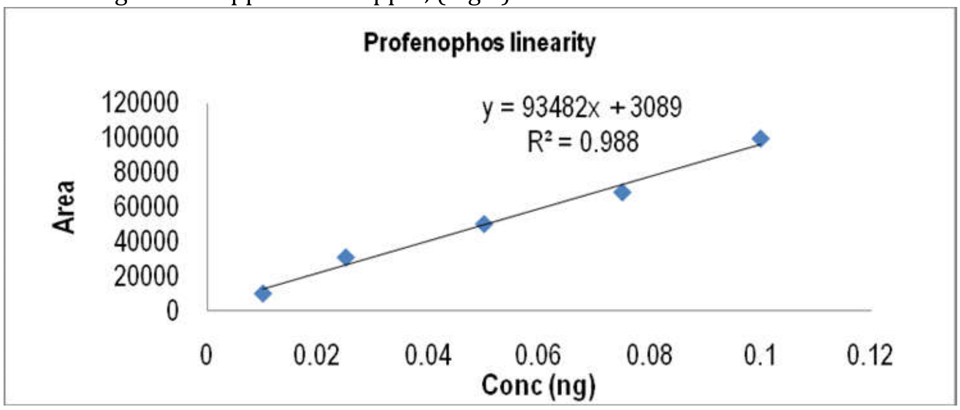

Fig 1. Calibration curve between concentration of profenophos versus Gas chromatograph peak area depicting linearity of response

Method validation

Prior to pesticide application and field sample analysis, the residue analysis method was validated following the principles as per SANCO document (12495 / 2011). 5 Kg of tomato fruits collected from untreated control plots were collected and the stalks were removed prior to samples preparation. The sample was homogenized using Robo Coupe Blixer and homogenized sample of each 15 g was taken in to 50 ml centrifuge tubes. The required quantity of profenophos intermediary standard prepared from CRM was added to each 15 g sample to get fortification levels of 0.05 ppm and 0.10 ppm in three replications each. These foritifcation levels are selected to know the suitability of the method to detect and quantify profenophos in tomato below Maximum Residue Limits (MRLs) of Codex Alimentarius Commission. The MRL of profenophos in tomato was 10 mg kg^{-1}, and 0.3 mg kg^{-1}, respectively.

The AOAC official method 2007.01 (Pesticide Residues of Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate) was slightly modified to suit to the facilties available at the laboratory and the same was validated for estimation of LOQ (Limit of Quantitation) of profenophos in Tomato matrix.

The recovery per centage and recovery factors was calculated using the following formula.

$$\text{Per cent Recovery} : \frac{\text{Residue quantified in fortified sample}}{\text{Fortified level}} \times 100$$

$$\text{Recovery factor} : \frac{100}{\text{Per cent recovery}}$$

Limit of Quantification (LOQ)

The fortified samples (0.05 and 0.10 mg kg^{-1}) were analysed as per the method described and the recovery factors were calculated. Tomato samples fortified with profenophos at 0.05 mg $kg^{-1}$and 0.10 **mg** $\mathbf{kg^{-1}}$ were analysed and the mean recovery of the residues using the method was 89.33% and 92.33%, respectively. The fortification and recovery results are presented in Table 2

Table 2. Recovery results of profenophos residues on tomato

Details	**Recoveries of profenophos from fortified tomato samples**			
	Fortified level (mg kg^{-1})			
	0.05 mg kg^{-1}		**0.10** mg kg^{-1}	
	Residues recovered (mg kg^{-1})	Recovery %	Residues recovered (mg kg^{-1})	Recovery %
R1	0.044	88.00	0.091	91.00
R2	0.046	92.00	0.094	94.00
R3	0.044	88.00	0.092	92.00
Mean		89.33		92.33
SD		2.309		1.527
RSD		2.585		1.654

Dissipation pattern of profenophos on tomato

Samples of tomato were collected from the plot treated with two sprays of profenophos 50 EC @ 1000 g a.i. ha^{-1} at regular intervals i.e. 0, 1, 3, 5, 7, 10, 15, 20 days after last spray, and analysed for residues and dissipation pattern of the insecticides was calculated.

RESULTS AND DISCUSSION

Dissipation of profenophos in tomato

The residue data of profenophos at 0, 1, 3, 5, 7, 10 and 15 days after second spray are presented in Table 3 Figure 2. and chromatograms in Figures 3, 4, 5 and 6.

Initial deposits of 1.16 mg kg^{-1} of profenophos detected at 2 hours after last spray, dissipated to below determination level (BDL) of 0.05 mg/kg by 7th day after last spray. The initial deposits dissipated to 0.67, 0.51 and 0.15 mg/kg by 1, 3, and 5 days after last spray, respectively. The dissipation pattern showed decrease of residues from first day to 5th day 42.24, 56.03 and 87.06% by 1, 3, and 5 days, respectively.

Table 3. Dissipation of profenophos in tomato

Days after last spray	**Residues of profenophos (mg kg^{-1})**	**Dissipation %**
	Average	
0	1.16	0
1	0.67	42.24
3	0.51	56.03
5	0.31	73.27
7	BDL	100
10	BDL	100
15	BDL	100
Regression equation	Y = 0.991 + (-0.144) X	
R^2	0.914	
Half-life	4.81 days	
Safe waiting period (MRL = 10 mg kg^{-1})	1 day	
BDL : Below Determination Level (< 0.05 mg kg^{-1})		

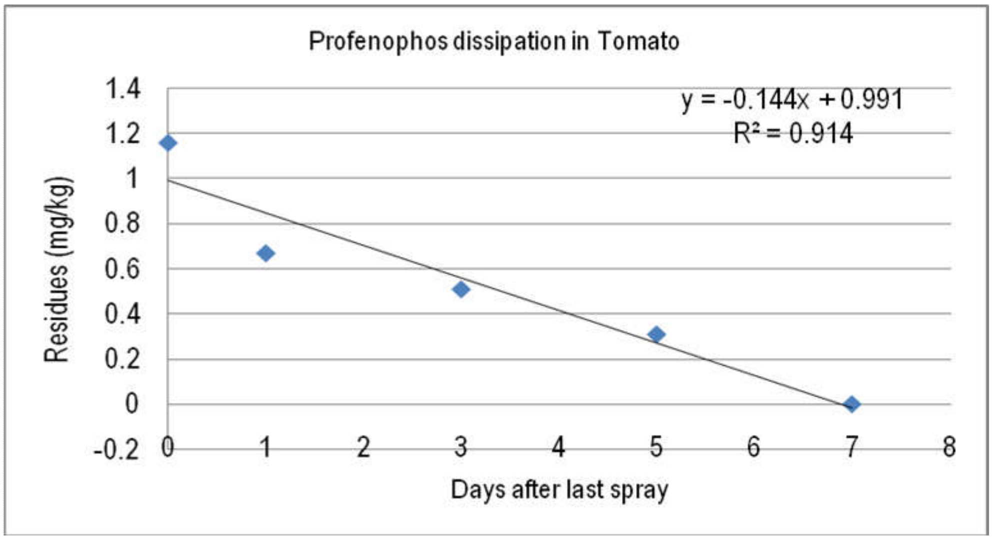

Fig. 2 Dissipation of profenophos in tomato

The regression equation was Y = 0.991 + (-0.144) X with R^2 of 0.914. Maximum residue limit for profenophos in tomato as per Codex Alimentarius Commission (CAC) and European Union (EU) is 10 mg kg^{-1}, and the suggested safe waiting period is 1 day, as the initial deposits are less than the MRL. The half life of profenophos on tomato was 4.81 days.

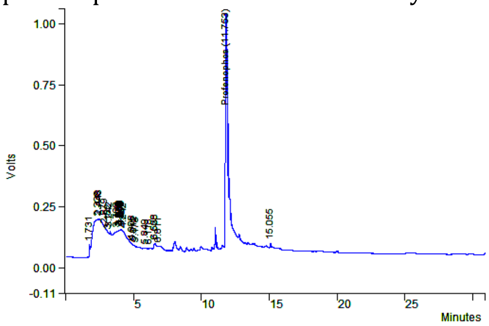

Fig.3. Chromatogram of profenophos in zero day sample

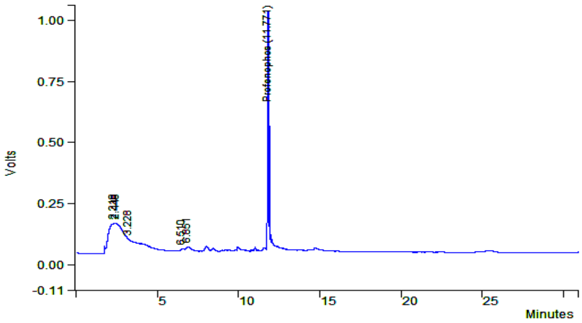

Fig.4. Chromatogram of profenophos in one day sample

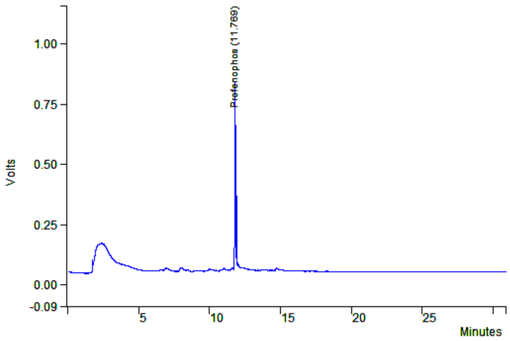

Fig.5. Chromatogram of profenophos in three day sample

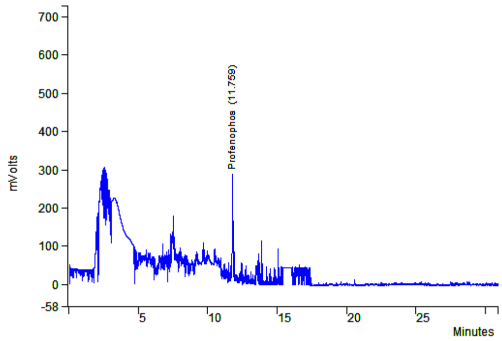

Fig.6. Chromatogram of profenophos in five day sample

The results are in agreement with the findings of Sahoo *et al.* [7] who reported that profenophos sprayed on tomato at 500 and 1000 g a.i. ha^{-1} at 50% flowering stage and subsequently at 15 days intervals does not seem to pose any hazards to the consumers with a waiting period of 3 days.

Helalia *et al*. [2]observed residues of profenophos in unwashed tomato fruits reached 0.643 ppm for profenophos by seven days after application. Shiboob [3] carried experiment to study the persistence pattern of profenophos in tomato and recorded safety time to be 10 days.

Romeh *et al*. [4] reported that tomato fruits can be safely harvested for human consumption or for processing purpose 7 days after spray in case of profenophos.

Abdalla *et al*. [5] experimented on tomato and *Phaseolus vulgaris* by spraying of profenophos, pirimiphos-methyl and methamidophos at recommended doses and reported that, tomatoes were considered to be safe for human consumption 1 day after treatment with pirimiphos-methyl and 8 days after treatment with profenofos or methamidophos where as *P. vulgaris* was considered safe 8 and 11 days after treatment with profenofos and pirimiphos-methyl, respectively. Experimental results of Radwan *et al.* [6] also shown that a waiting period of 10 and 14 days was worked out from the data generated in field sprayed profenophos @ 400 g a.i. ha-1 on green pepper and eggplant, respectively.

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