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Research article

DISSIPATION PATTERN OF BIFENTHRIN IN TOMATO

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ABSTRACT: Field experiment carried out during *kharif*, 2012 to evaluate the dissipation pattern of most commonly used insecticide bifenthrin 10 EC @ 100 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 bifenthrin were 0.85 mg kg^{-1} which dissipated to 0.39, 0.15 mg kg^{-1} by 1st and 3rd day after last spray, respectively, and to BDL by 5th day. Bifenthrin initial deposits were dissipated to 82.35% by 3rd day after last spray, and pre-harvest interval of 3 days is suggested taking into consideration of MRLs of bifenthrin in tomato (0.3 mg kg^{-1}) as per Codex Alimentarius Commission.

Key words: Tomato, dissipation pattern, Bifenthrin

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, tomato 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} (NHB 2011). 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, the studies conducted to evaluate the bio-efficacy of certain most commonly used insecticide and also residue dynamics of bifenthrin 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 further chemical dissipation studies was carried out .

Pesticide Residue Analysis Method

Preparation of working standards of bifenthrin

Certified Reference Materials (CRMs) of bifenthrin purchased from Dr. Erhenstorfer, Germany. Primary, intermediary and working standards were prepared from the CRMs using acetone and hexane as solvents. Bifenthrin 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 bifenthrin were injected in Gas Chromatograph VARIAN GC 3800 with Electron Capture Detector (ECD) and Thermionic Specific Detector (TSD) for estimating the lowest quantity of bifenthrin which can be detected with injector split ratio of 1:10 under standard operating parameters as given below. For confirmatory analysis, bifenthrin 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 bifenthrin 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)	Bifenthrin 16.03 min
Total run time (min)	30 min

Under the GC operational parameters given in Table 1. the retention time of bifenthrin 16.03 min, respectively.
Each working standard of bifenthrin (0.05 ppm, 0.075 ppm, 0.10 ppm, 0.25 ppm, 0.50 ppm and 0.75 ppm) were also 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 bifenthrin is 0.05 ng, and the linearity is in the range of 0.05 ng to 0.75 ng as given in Fig 1.

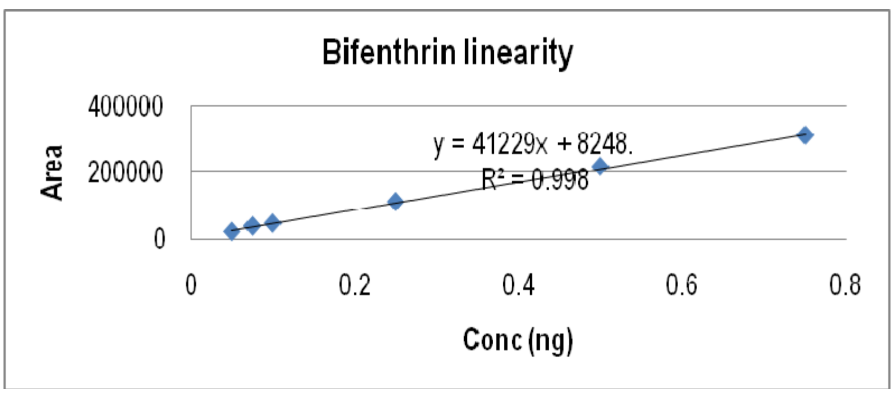

Fig 1. Calibration curve between concentration of bifenthrin Versus Gas chromatograph peak area depicting linearity of response

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). 5Kg 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 bifenthrin 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 bifenthrin in tomato below Maximum Residue Limits (MRLs) of Codex Alimentarius Commission. The MRL of bifenthrin in tomato was 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 bifenthrin in Tomato matrix.
The recovery percentage and recovery factors were 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 bifenthrin at 0.05 mg $kg^{-1}$and 0.10 mg kg^{-1}were analysed and the mean recovery of the residues through the method was 88% and 92.66%, respectively.The fortification and recovery results are presented in Table 2.

Table 2. Recovery results of bifenthrin residues on tomato

Details	Recoveries of bifenthrin 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	0.043	86.00	0.092	92.00
R2	0.044	88.00	0.095	95.00
R3	0.045	90.00	0.091	91.00
Mean		88.00		92.66
SD		2.000		2.081
RSD		2.272		2.246

Dissipation pattern of bifenthrin on tomato

Samples of tomato were collected from the plot treated with two sprays of two sprays of bifenthrin 10 EC @ 100 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 bifenthrin in tomato

The residue data of bifenthrin at 0, 1, 3, 5, 7, 10 and 15 days after spraying @ 100 g $ai.ha^{-1}$ are presented in Table 3, Figure 2 and chromatograms are presented in figures 3, 4 and 5. Initial deposits of 0.85 mg kg^{-1} of bifenthrin detected at 2 hours after last spray, dissipated to Below Determination Level (BDL) by 5th day after last spray. The initial deposits dissipated to 0.39 and 0.15 mg kg^{-1} at 1 and 3 days after last spray, respectively. The dissipation pattern showed decrease of residues from first day to 3rd day 54.11 and 82.35% by 1st and 3rd day, respectively.

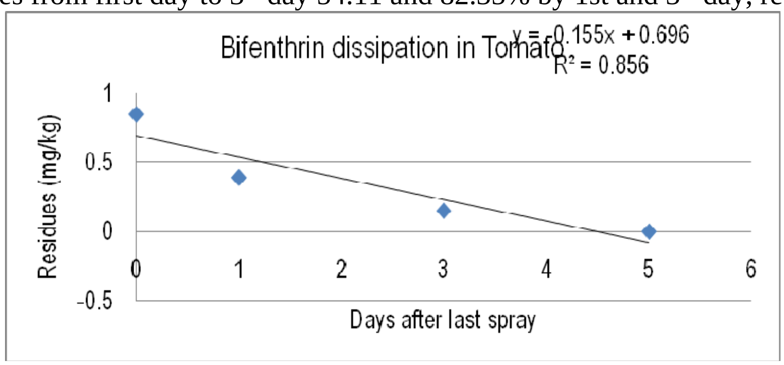

Fig. 2 Dissipation of bifenthrin in tomato

Table 3. Dissipation of bifenthrin in tomato

Days after last spray	Residues of bifenthrin (mg kg^{-1}) Average	Dissipation %
0	0.85	0
1	0.39	54.11
3	0.15	82.35
5	BDL	100
7	BDL	100
10	BDL	100
15	BDL	100
Regression equation	Y = 0.696 + (-0.155) X	
R^2	0.856	
Half-life	4.47 days	
Safe waiting period (MRL = 0.30 mg kg^{-1})	2.358 days (= 3 days)	
BDL : Below Determination Level (<0.05 mg kg^{-1})		

The regression equation is Y = 0.696 + (-0.155) X with R^2 of 0.856. Maximum Residue Limit for bifenthrin in tomato as per Codex Alimentarius Commission (CAC) and European Union (EU) is 0.30 mg kg^{-1}, and the calculated safe waiting period is 2.358 days, and the suggested safe waiting period is 3 days. The half life of bifenthrin on tomato was 4.47 days.

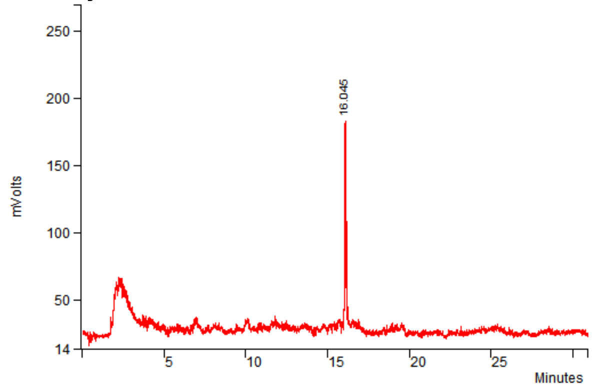

Fig.3. Chromatogram of bifenthrin in zero day sample

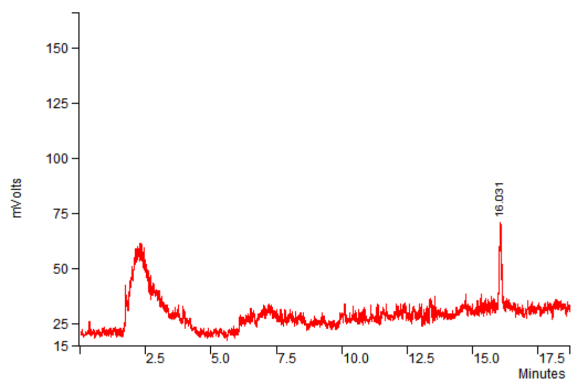

Fig.4 . Chromatogram of bifenthrin in one day sample

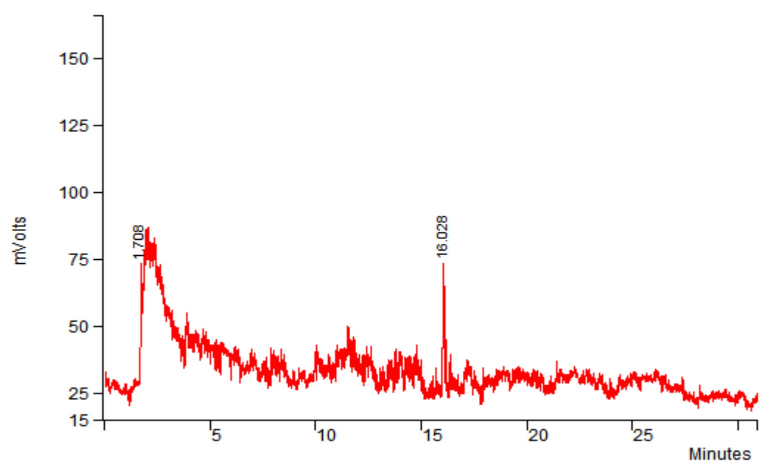

Fig.5. Chromatogram of bifenthrin in three day sample

Reena Chauhan *et al*. (2012) studied the behaviour of bifenthrin on tomato crop following the application of 25 g a.i ha-1 and 50 g a.i ha^{-1} and found that the residues were reached below detectable level of 0.005 mg kg^{-1} on 10^{th} day after application. Similarly, Suman Gupta *et al*. (2009) reported that the bifenthrin residues were persistent up to 10 days with half-life of 1.32-1.58 days when applied @ 25 and 50 g a.i. ha^{-1}, and suggested waiting period of 1 day for bifenthrin on tomato. Bouri *et al*. (2012) calculated a safe waiting period of 4 days for bifenthrin in green bean in the winter and 3.5 days in the spring. Balakrishnan *et al*. (2009) reported that the harvest time residues of bifenthrin were at below detectable level in lint, seed, oil and soil samples of cotton when applied 10 EC @ 80 and 160 g a.i. ha^{-1}. The dissipation of pesticide residues in/on crops depends on climatic conditions, type of application, plant species, dosage, interval between application and time of harvest (Khay *et al.*, 2008). Salghi *et al*. (2012) analyzed residues of 8 pesticides in tomato samples and found residue levels ranged from 0.001 to 0.400 mg kg^{-1} for dicofol, from 0.003 to 0.170 mg kg^{-1} for procymidone, from 0.001 to 0.250 mg kg^{-1} for chlorothalonil, 0.050 to 0.500 mg kg^{-1} for bifenthrin, 0.001 to 0.010 mg kg^{-1} for lambda -cyhalothrin, 0.001 to 0.300 mg kg^{-1} for cypermethrin, 0.010 to 1 mg kg^{-1} for deltamethrin and from 0.003 to 1.123 mg kg^{-1} for endosulfan. You Xiang *et al*. (2013) carried out experiments to check the residue levels of bifenthrin in wheat and observed the half-lives of bifenthrin in wheat seedlings ranged from 2.4 to 10.5 days and at harvest time, the terminal residues of bifenthrin were below the maximum residue limit (0.5 mg kg^{-1}) set by Codex Alimentarius Commission. Hajslova *et al*. (1997) determined the residues of 6 widely popular pesticides *viz.*, alphamethrin [alpha-cypermethrin], bifenthrin, cyhalothrin, deltamethrin, fluvalinate and permethrin in cereals and found all the insecticides in the detection limits ranged from 0.0002 to 0.005 mg kg^{-1}.

Patyal *et al*. (2010) studied persistence of bifenthrin at @ 60 g a.i. ha^{-1} and 120 g a.i./ha on apple fruit and observed the residues of bifenthrin initially ranging from 0.669-1.062 mg kg^{-1} at 60 g a.i./ha dose and 1.348-1.784 mg kg^{-1} at 120 g a.i. ha^{-1} dose which reduced to half in 4.85-5.22 and 4.38-6.66 days at the respective doses and suggested safe waiting period of 2.1-5.4 and 6.7-11.3 days, based on the Maximum Residue Limit (MRL) value of 0.5 mg kg^{-1} of bifenthrin on apple. Soudamini and Sharma. (2007) studied the persistence of bifenthrin (Talstar 10 EC) on mango with 3 spray applications at recommended (0.5 ml l^{-1}) and double dose (1.0 ml l^{-1}) to found that the initial bifenthrin deposits of 0.82 and 1.45 µg g^{-1} and half-life of bifenthrin on mango peel was 11 days. Based on the maximum residue limit (MRL) value of 0.5 µg g^{-1}, a pre-harvest interval of 6 and 16 days are suggested for recommended and double the recommended doses. Tewary *et al*. (2005) studied the dissipation behavior of bifenthrin in tea and reported that a waiting period of at least three days before plucking the tea shoots after pesticide application at recommended dose (40 g a.i. ha^{-1}).

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