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Dissipation of combination product spirotetramat 120 SC + imidacloprid 120 SC (Movento Energy) in/on tomato

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Abstract

Studies on the dissipation pattern of spirotetramat and imidacloprid in/on tomato fruits were undertaken by following three foliar applications at recommended and double the recommended doses of combination product spirotetramat 120 SC + imidacloprid 120 SC @ (75 and 150 g a.i./ha) at fruiting stage. Residues of spirotetramat and imidacloprid dissipated with half-life of 1.93, 1.37 and 1.40, 1.92 days, at both the doses respectively. The residues reached below quantification limit (BQL) on 5th day in spirotetramat and 7th day in imidacloprid at the recommended dose. Considering this, Pre-harvest interval (PHI) of 7 days can be suggested for combination product with reduced risk of insecticide residues in tomato.

Keywords: Spirotetramat, imidacloprid, persistence and QuEChERS

Introduction

Tomato (*Solanum lycopersicon* Miller.) is one of the most important and remunerative vegetable crop grown in tropical and subtropical region of the world for fresh market and processing, constituting an important part of our human diet. Tomato rank first among the processed vegetables. It is a very good source of income to small and marginal farmer. Globally, tomato is cultivated over an area of 4.8 million ha with annual production of 282,830 million MT with the productivity of 37.66 MT ha⁻¹ (Anon., 2018) [1]. In India, tomato is mainly grown in *kharif* and *rabi* seasons across the country whereas in some regions it is produced throughout the year. It occupies an area of about 0.78 million ha producing over 19.37 million MT with productivity of 24.65 MT ha⁻¹. In Maharashtra approximately 43640 ha area is covered under tomato with a production of 0.95 million MT with average productivity of 21.93 MT⁻¹ (Anon., 2018) [1]. Tomato, like other vegetables, is prone to insect pests and disease mainly due to tenderness and softness as compared to other crops. The tomato yield in India is considerably lower because of several factors of which damage caused by insect pests is most important. It is devastated by an array of pests like jassids, aphids, tobacco caterpillar, flea beetles, spider mites, and fruit borer. However the major economic damage is caused by the fruit borer. Tomato fruit borer, *Helicoverpa armigera* (Hubner) (Lepidoptera: Noctuidae) is most destructive polyphagous and assumed a status of 'key pest' in all part of world. It feeds and breeds on 181 species of host plant (Manjunath *et al.* 1989) [5].

Commercial consideration of this crop unfortunately compelled the growers to use a large amount of pesticides during the entire period of growth even at fruiting stage and sometimes farmers also ignored the recommended waiting period between the harvest and last spray. In some cases the residues of insecticide exceeded its tolerances. The degradation or dissipation of insecticide is influenced by climatic condition, type of application, plant species, dosage, interval between last application and time of harvest (Khay, *et al.*, 2008) [3]. Therefore, it is necessary to study the safe waiting period for consumption of tomato fruits and persistence of introduced pesticides in crop.

Material and Methods Field experiment

The experiment was laid out at the Instructional Farm of Post Graduate Institute, M.P.K.V, Rahuri during *Kharif-2019*.

Tomato seedlings were grown on raised beds by sowing disease free seeds of variety 'Meghdhoot'. Seedlings were ready for transplanting on 30th days after sowing. Seedlings transplanted having spacing 60 x 45 cm. Overall three foliar sprays of insecticide was given at an interval of 10 days starting at fruit initiation stage, two doses of spirotetramat (recommended dose 75 g a.i.ha-1 and double the recommended dose 150 g a.i.ha-1) and two doses of imidacloprid (recommended dose 75 g a.i.ha-1 and double the recommended dose 150 g a.i.ha-1) were evaluated for residues.

Chemicals and reagents

The certified reference material of spirotetramat, spirotetramat-enol and imidacloprid with purity of 99.2%, 99.6% and 97.2% were obtained from Sigma Aldrich and commercial formulations (Movento energy 240 SC) were purchased from local market of rauri. HPLC grade acetonitrile and Analytical reagent grade sodium acetate was obtained from M/s. Avantor performance Material India Limited, Thane. PSA and Magnesium sulphate was procured from Agilent Technology, Bangalore and RFCL, Gujarat, respectively. Working standards were prepared by dissolving reference standards in acetonitrile.

Residues analysis standard preparation

An accurately weighed 10 mg of an individual analytical grade insecticide was dissolved in 10 ml volumetric flask using suitable solvent to prepare the standard stock solution of 1000 mg kg-1. Standard stock solution of each insecticide was further diluted to obtain intermediate lower concentration of 100 and 10 mg kg-1. They were stored in a refrigerator at -40°C. From intermediate standards, were prepared by suitably diluting the stock solution in acetonitrile for spirotetramat, spirotetramat-enol, imidacloprid and was used as standard check in analysis, linearity and recovery studies.

Method validation

Prior to analysis of samples, linearity of spirotetramat, spirotetramat-enol, imidacloprid was established on HPLC. Accuracy and precision of the method was determined by per cent mean recovery and percent relative standard deviation (RSD). The limit of detection (LOD) of spirotetramat, spirotetramat-enol, imidacloprid was determined by considering a signal-to-noise ratio of three with reference to the background noise obtained for the blank sample. The limits of quantification (LOQ) determined as 3 times of LOD. Tomato fruits sample (15 g) was taken in 50 ml centrifuge tubes in three replicates each was spiked with spirotetramat, spirotetramat-enol, imidacloprid separately at the required fortification levels i.e. LOQ, 5 x LOQ and 10 x LOQ, adding an appropriate volume of working standard of 10 mg kg-1. The extraction and clean-up was performed using methodology as described under. The per cent recovery was calculated by using following formula

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

Residue determination

Residue of spirotetramat, spirotetramat-enol, imidacloprid was performed using HPLC. Identification of insecticide residue was accomplished by retention time and compared with known standard at the same condition. The quantities were calculated on peak area basis by using following formula.

Residues (mg kg ⁻¹)	Area of Sample	X	µl of sample injected	X	conc. of standard (ppm)	X	Final Vol (ml)
	Area of standard		µl of standard injected		Wt. of sample (g)		

$$\text{Wt. of sample (g)} = \frac{\text{Sample Wt. (g)} \times \text{Aliquot taken (ml)}}{\text{Volume of solvent added (ml)}} = \text{g}$$

Statistical analysis

The simple statistical analysis was carried out in the Microsoft Excel programmed with the help of computer. The mean residues, standard deviation, regression equation, R² value and half life values were calculated in excel programme.

Sample collection

The medium marketable size tomato fruit samples (1 kg) were collected from each plots 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 samples (tomato fruits) were transferred immediately to the laboratory in an ice box. The collected tomato samples were brought to the laboratory in polythene bags and processed immediately.

Extraction and cleanup

The tomato samples were extracted and cleaned up using modified QuEChERS method (Quick, Easy, Cheap, Effective, Rugged and Safe (Sharma, 2013) ^[10]). The entire laboratory sample was crushed thoroughly in grinder and approximately 15 g homogenized sample weighed in a 50 ml polypropylene tube and tube was kept in deep freezer for 10 min. to this, 15 ml of 1% acetic acid in acetonitrile, 6.0 g MgSO₄ and 15 g sodium acetate. It was shake vigorously for 1.0 min (manually). Then centrifuged at 3500 rpm for 1 min and transfer 6 ml supernatant to the 15 ml tube containing 300 mg PSA + 900 mg MgSO₄ and shake vigorously for 30 sec. After this centrifuged at 3500 rpm for 1.0 min to separate the solid materials. Two ml of supernatant was taken and evaporated to dryness and made up to 2.0 ml with acetonitrile HPLC analysis.

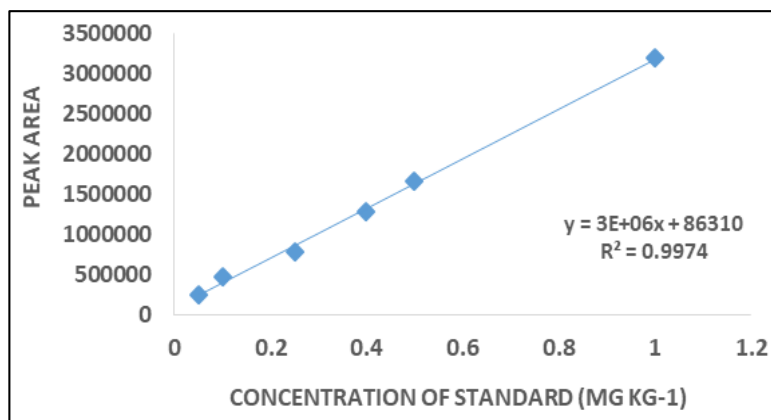
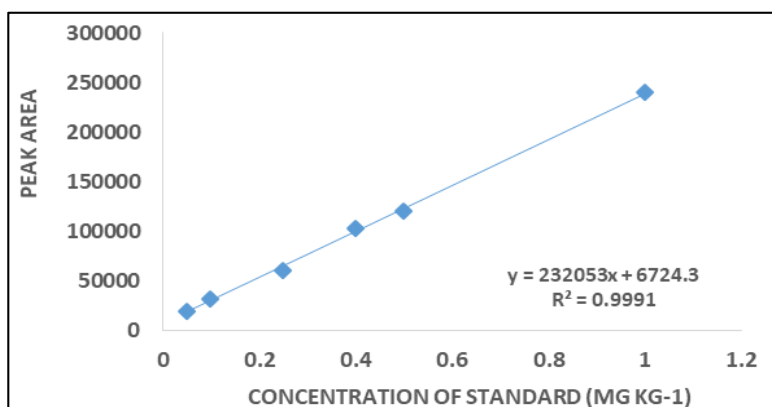
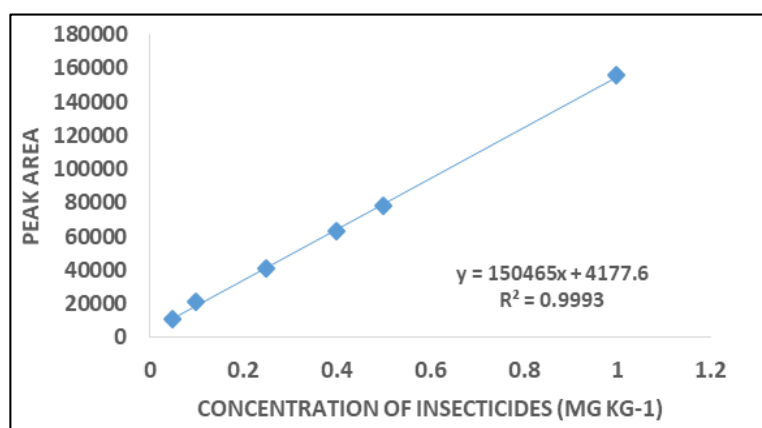
Estimation

HPLC parameter

The analysis of samples of imidacloprid and spirotetramat residues was carried out with Shimadzu make high-performance liquid chromatography system equipped with diode array detector (HPLC) and quaternary pump (LC-20 AT). LC solution software was used as the data analysis system. The operating parameters of the instruments are shown below:

Table 1: HPLC parameter

Column type	:	Purospher @ STAR (Hibar) RP-18(5u) m-150-4.6
Mobile phase	:	Acetonitrile: Water (80:20)
Flow rate	:	0.8 ml min ⁻¹
Wavelength	:	254 nm
Injector volume	:	20 µl
Retention time	:	Imidacloprid 3.21 min Spirotetramat 4.39 min Spirotetramat-enol 3.46 min

**Fig 1:** Spirotetramat linearity**Fig 2:** Spirotetramat-enol linearity**Fig 3:** Imidacloprid linearity**Table 2:** Mean recovery of different insecticides in tomato

Substrate	Fortification level (mg/kg)	Recovery (%)		
		Spirotetramat	Spirotetramat-enol	imidacloprid
Tomato fruits	0.05	90.47	89.55	89.91
	0.25	94.00	93.04	94.02
	0.50	93.17	93.64	94.90

Result and Discussion

Persistence and Dissipation of Spirotetramat in/ on Tomato Fruits

Dissipation of residues in plant depends on climatic condition, type of application, dosage and interval between application and time of harvest. The results revealed reduction in residue level of these tested insecticides in tomato fruits with time.

The overall result of analysis of tomato fruit following three applications of Spirotetramat + imidacloprid 120 SC @ 75 and 150 g a.i.ha⁻¹ are presented in (Table. 3). The mean initial deposits of spirotetramat were 0.57 and 1.20 mg kg⁻¹ on tomato fruits. These deposits dissipated to 0.27 and 0.59 mg kg⁻¹ after 1 day at recommended and double the recommended dosages, respectively, thereby showing a loss of more than 50%.

The residue dissipated to 73.68 and 93.33 per cent at both the doses of application (Table 3 and Fig.4). Half-life value calculated for spirotetramat on tomato was 1.93 and 1.37 days for recommended and double the recommended dose, respectively. The residue of spirotetramat reached below its limit LOQ of 0.05 mg kg⁻¹ in 5 and 7 days at recommended and doubles the recommended dosages, respectively. Metabolite of spirotetramat i.e. spirotetramat-enol, was not detected in any of the sample.

The present findings are in agreement with Chahil *et al.*, (2014) [2] who studied dissipation of siprotetramat in green chilli fruits and soil at 120 and 240 g a.i.ha⁻¹, recommended and double the recommended dose. The initial deposits were recorded as 0.55 and 1.22 mg kg⁻¹ for both doses, respectively, which reached below detection limit in 5th and 7th day at recommended and double recommended dose, respectively.

The half-life values calculated were 1.91 and 1.30 at recommended and double recommended dose, respectively. Similar type of study was conducted by Pandiselvi *et al.*, (2010) [8] studied the residues of spirotetramat in cotton plant, seed, lint and oil @ 90 and 180 g a.i.ha⁻¹ recommended and double the recommended dose. The initial residue of spirotetramat on cotton plant were found to be 0.04 and 0.08 mg kg⁻¹ at recommended and double recommended dose, respectively.

These residue were found to be below determination limit at 3 and 5 days, respectively. Cotton seed, lint, and oil samples collected at the time of harvest showed no detectable residues of spirotetramat. Mohapatra *et al.*, (2012) [6] studied the persistence of spirotetramat in mango fruits following application of spirotetramat at 90 and 180 g a.i.ha⁻¹ and

reported that the residues were found to be below determination limit at 10 days for both dosages.

Persistence and dissipation of imidacloprid in/ on tomato fruits: In case of imidacloprid 120 SC @ 75 and 150 g a.i.ha⁻¹ the average initial deposits of imidacloprid on tomato fruit were found to be 0.76 and 1.52 mg kg⁻¹, respectively, following three application of combination mixture of spirotetramat 120 SC + imidacloprid 120 SC with respect to imidacloprid at 10 days interval. These deposits dissipated to 0.45 and 0.93 mg kg⁻¹ after 1 day at recommended and double the recommended dosages, respectively.

More than 65% of these residues got dissipated in the third days at both the dosage. The residue dissipated to 92.10 and 95.39 percent at both the doses of application (Table 4 and Fig. 5). Half-life value calculated for spirotetramat on tomato was 1.40 and 1.92 days for recommended and double the recommended dose, respectively. The residue of imidacloprid reached below its limit LOQ of 0.05 mg kg⁻¹ in 7th and 10th days at recommended and double the recommended dosages, respectively. The results obtained were found to be in agreement with Chahil *et al.*, (2014) [2] reported the persistence of imidacloprid in chilli fruits by following three application of a mixture formulation of imidacloprid at 1000 and 2000 mL ha⁻¹. The initial residue reached below detectable limit on 7th and 10th and Half-life periods were observed to be 1.41 and 1.65 days at recommended and double the recommended dosages, respectively. Nasr (2014) [7] reported the residues on cucumber fruits collected after 1 hr, 1, 3, 5, 8, 11, 15 and 21 days from last spray and observed the residue half-life (RL₅₀) value of imidacloprid is 2.2 days.

Similar type of study was conducted by Varghees, (2014) [11] who reported the dissipation of neonicotinoid insecticide, imidacloprid on chilli fruits drawn at 0, 1, 3, 5, 7, 15, 21 days after spraying with the half-life (RL₅₀) vale of 2.08 days.

Similar type of study conducted Mandal *et al.*, (2010) [4] who reported the dissipation of imidacloprid on brinjal following three application of a combination formulation of Solomon 300 OD (β- cyfluthrin 9% + imidacloprid 21%) at 42 and 84 g a.i.ha⁻¹. Initial deposits were 0.24 and 0.37 mg kg⁻¹ at single and double dose, respectively. Imidacloprid residues took 10 days to reach LOQ at both dosage. Sahoo *et al.*, (2012) [9] studied the imidacloprid residues on okra fruits following application of Solomon 300 OD at 200 and 400 mL ha⁻¹ which dissipated to below detectable level after 5th and 7th days at single and double the dosages.

Table 3: Dissipation pattern of spirotetramat in tomato

Interval between last application and sampling	Spirotetramat			
	Recommended dose @ 75 g a.i. ha ⁻¹		Double the Recommended dose @ 150 g a.i. ha ⁻¹	
	Mean Residue (mg/kg)	Dissipation (%)	Mean Residue (mg/kg)	Dissipation (%)
0 day	0.57	--	1.20	--
1 day	0.27	52.63	0.58	51.66
3 day	0.15	73.68	0.35	70.83
5 day	BQL	--	0.08	93.33
7 day	BQL	--	BQL	--
10 day	BQL	--	BQL	--
15 day	BQL	--	BQL	--
RL ₅₀ (days)	1.93		1.37	

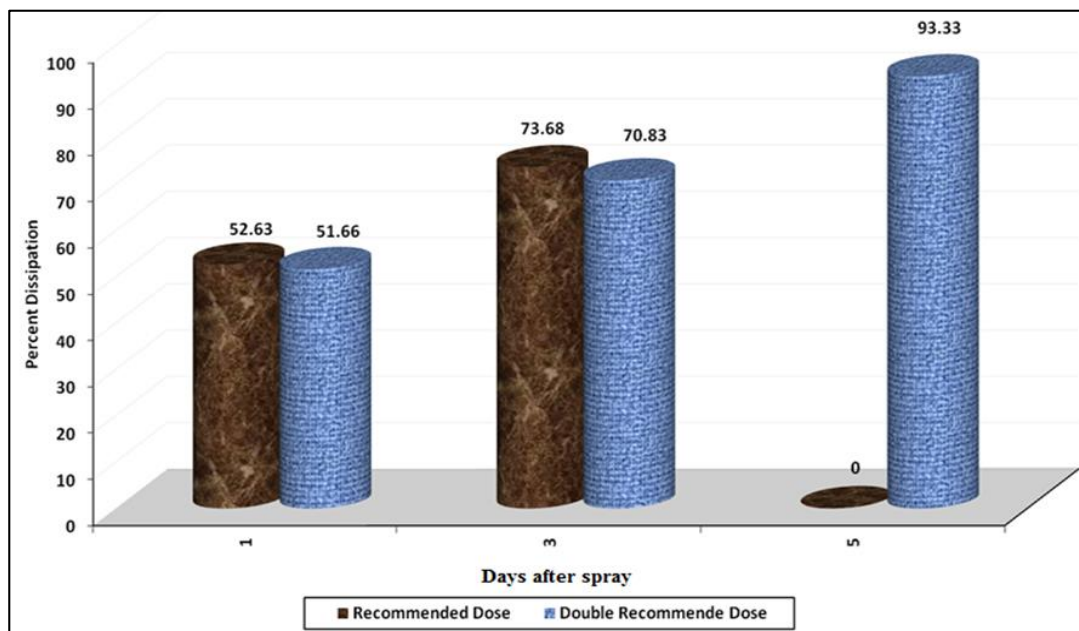


Fig 4: Percent dissipation of spirotetramat in tomato fruit

Table 4: Dissipation pattern of imidacloprid in tomato

Interval between last application and sampling	Imidacloprid			
	Recommended dose @ 75 g a.i. ha ⁻¹		Double the Recommended dose @ 150 g a.i. ha ⁻¹	
	Mean Residue (mg/kg)	Dissipation (%)	Mean Residue (mg/kg)	Dissipation (%)
0 day	0.76	--	1.52	--
1 day	0.45	40.78	0.93	38.81
3 day	0.22	71.05	0.54	64.47
5 day	0.06	92.10	0.33	78.28
7 day	BQL	--	0.07	95.39
10 day	BQL	--	BQL	--
15 day	BQL	--	BQL	--
RL ₅₀ (daya)	1.40		1.92	

BQL = Below Quantification Limit

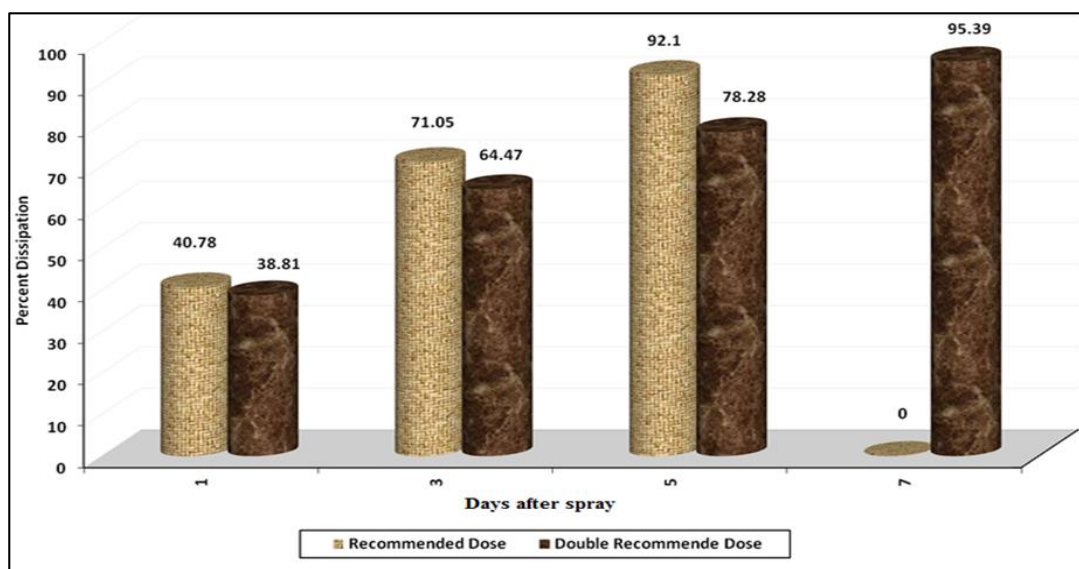


Fig 5: Percent dissipation of imidacloprid in tomato fruit

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