Trends in Biosciences 10(43), Print : ISSN 0974-8431, 8967-8970, 2017

Harvest Time Residues of Thiamethoxam 25% WG in Mango

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ABSTRACT

Studies were conducted to evaluate the harvest time residues of thiamethoxam 25% WG on mango in Agricultural Research Station (ARS), Bhavanisagar of Erode district during 2013. The treatments comprised of thiamethoxam 25% WG at 25 and 50 g a.i. ha^{-1} along with an untreated check. Samples of mango fruits and soil were collected in a random manner at harvest for analysis. The samples were extracted with QuEChERS method and subjected to analyzed by using Gas chromatography equipped with Electron Capture Detector. The retention time of thiamethoxam 25 % WG was 10.52 minutes with recovery ranged between 83.12 to 89.23 and 81.14 to 88.64 for mango fruit and soil, respectively. The harvest time residues of thiamethoxam 25% WG at 25 and 50 g a.i. ha^{-1} were below detectable level in fruit and soil.

Key word *Thiamethoxam 25% WG, Mango, residue, harvest time, soil*

Mango (*Mangifera indica* Linn.) referred to as the "King of fruits" is one of the major fruit crops of South Asia from time immemorial and still remains a prominent horticultural crop of India. It has enormous potential in fruit processing industries and in foreign exchange apart from local consumption. Mango is usually attacked by pests like hoppers and mealybugs. Among the insect pests, hoppers are considered to be the most destructive pests affecting the quality of mangoes. Yield loss due to hopper was estimated to be about 25 to 60 per cent (Ray *et al.,* 2008). During the last few decades, chemical control using insecticides was the most efficient method to minimize sucking pest damage in crop production. The repeated application of synthetic pyrethroids led to the resurgence of mango hoppers (Sushil kumar *et al.,* 2005). Indiscriminate and consistent use of insecticides leads to environmental pollution, hazards to beneficial fauna and flora, insecticidal resistance, pest resurgence and sudden outbreak of pest species. Moreover, people are also exposed to pesticides not only through ingestion of contaminated fruits and vegetables grown, but while applying these in the field. The second generation neonicotinoid, thiamethoxam is one of the most effective chemical exhibiting high insecticidal activities against homopteran pests as well as safe to natural enemies.

Pesticide residue analysis is essential to address rising consumer concerns regarding possible contamination issues. Several authors have dealt with the determination of thiamethoxam residues in various samples using different method *viz.,* GC (Gas chromatography) by using nitrogen phosphorus detector (Gabriela *et al.,* 2006), HPLC (High performance liquid chromatography) coupled with UV-VIS (Ultraviolet–visible spectroscopy) detector (Karmakar *et al.,* 2012). The QuEChERS (quick, easy, cheap, effective, rugged, and safe) method has received the distinction as an AOAC (A*ssociation of analytical communities)* official method for measuring multiple pesticide residues in fruits and vegetables (Lehotay, 2007). The present investigation was carried out to study the harvest time residues of Thiamethoxam 25% WG in mango.

MATERIALS AND METHODS

Field experiment was conducted at Agricultural Research Station **(**ARS), Bhavanisagar of Erode district during January to February 2013 to evaluate the residues of thiamethoxam 25 WG in ten year old Potalma variety of mango. The treatments comprised of thiamethoxam 25 % WG @ 25 and 50 g a.i ha^{-1}; an untreated control with three replications was simultaneously maintained during the study. The field experiments were laid out in randomized block design (RBD) with spacing of 10 m x 10 m.

For insecticide residue analysis, sampling of mango fruit was done during first harvest from the treatments after the last spraying. Soil samples were collected during first harvest from the treated and untreated plots by using a hand held auger driven to a plough depth of 15 cm. Control samples were collected from untreated plots. The samples were brought to laboratory in dry ice boxes and kept under -18 ^{0}C before analysis.

Residues analysis

Fruit

Mango fruits were processed by Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) method. Two fifty gram of samples were taken and blended in a blender. Samples weighing 10 gram was taken in 50 ml centrifuge tubes to which 20 ml of acetonitrile was added and shaked well. To this mixture, 4 g of MgSO4 and 1 g of sodium chloride were added, shaken vigorously by hand for one minute and the mixture was then centrifuged at 10,000 rpm for 10 minute. Six ml portion of the upper layer was transferred into a 25 ml of centrifuge tube containing 100 mg Primary and Secondary Amines (PSA) and 600 mg of MgSO4, The mixture was shaken vigorously by hand for 1 minute and then centrifuged for 10 minutes at 5000 rpm to separate solids from solution. Four ml of supernatant from the 25 ml of centrifuged tube was transferred to turbovap tube. The extract was condensed up to dryness in turbovap evaporator and the volume was made up to one ml by hexane (HPLC grade). The extract was stored in glass vial for final determination.

Soil

An air dried and seived soil sample (10 g) was taken in a 100 ml conical flask and mixed with 50 ml acetonitrile

and shaken for 1h in a mechanical shaker. The solution was filtered, condensed to near dryness and added 10 ml hexane and then filtered 1 ml through membrane filter and 20 ìl of this sample was directly injected to GC (Model Varian CP 3800), equipped with Electron Capture Detector.

Final quantification

Thiamethoxam 25% WG residues were estimated by Gas chromatography Varian CP 3800 equipped with Electron Capture Detector (ECD) fitted with Varian DB 5 Colum (length 30 m; diameter 0.25mm). The following were the operating parameters.

Column	Varian DB 5
Column Temperature	Variable temp. (180^0C, hold time 1.0 minute, ramp 250^0C @ 5^0C min^{-1} and hold time 2 minutes)
Injector Temperature	220^0C
Detector, Temp	Electron Capture Detector -300^0C
Carrier Gas	Nitrogen
Flow rate	1 ml min^{-1}
Total run time	20 minutes
LOD	0.003
LOQ	0.01

The amount of residue was determined by comparing the sample response with the response of standard by using the formula,

$$\text{Residue in ppm } (\mu g\ g^{-1}) = \frac{A_1 \times C \times V_1 \times V_{std}}{A_2 \times W \times V_s} \times R_f$$

Where,

A_1 = Area of compound from sample, in chromatogram

A_2 = Area of compound from standard, in chromatogram

V_1 = Total volume of sample in ml

C = Concentration of analytical standard in µg g^{-1}

W = Weight of the sample in g

V_{std} = Volume of the standard injected in µl

V_s = Volume of sample injected in µl

R_f = Recovery factor (if applicable)

RESULTS AND DISCUSSION

The endogenous compounds interfering with the analyst were assessed by comparing the chromatograms of the standard, blank sample and fortified sample. There were no interference peaks at the retention time of thiamethoxam which was at 10.52 min. The developed method consisted of salting out extraction and GC ECD detection and was very selective in enabling analysis of the analyte in the enormous matrix components. The recovery ranged between 83.12 to 89.23 and 81.14 to 88.64 for mango fruit and soil, respectively. The limit of quantification (LOQ) was 0.01 µg g^{-1} and the limit of detection (LOD) was 0.003 µg g^{-1}. The harvest time residues of thiamethoxam 25 WG sprayed thrice at 25 and 50 g a.i. ha^{-1} were below detectable level (BDL) in mango fruit and soil.

The present findings are in confirmation with the findings of several authors who reported that the harvest time residue of thiamethoxam was below detectable level in tomato and citrus (Karmakar and Kulshrestha, 2009, Malhat *et al.,* 2014). The fate of thiamethoxam in soil might be affected by a variety of complex factors such as pesticide properties, soil characteristics, environmental conditions, etc. Low residues in soil at harvest time would be largely due to the formulation sprayed directly on the leaves (Pateiro *et al.*, 2008). Thus the harvest time residues of thiamethoxam 25 % WG at 25 and 50 g a.i.ha^{-1} were found to be below the determination level and hence were safe.

Table 1. Recovery of thiamethoxam in mango

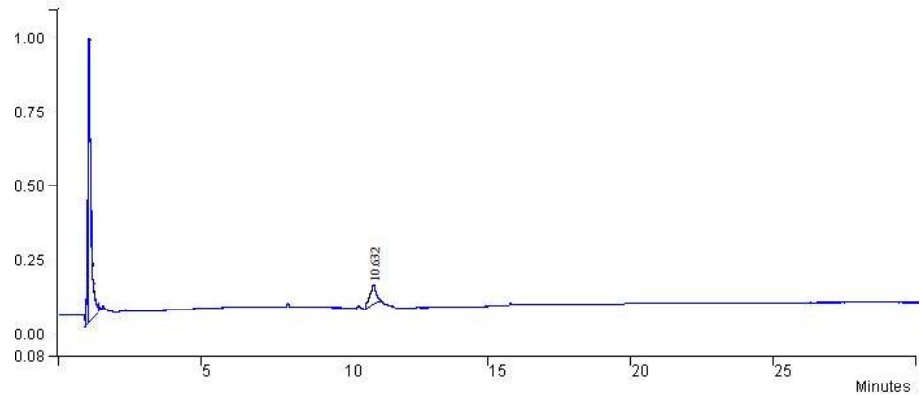
Crop	Matrix/substrate	Amount fortified (µg g^{-1})	% Recovered*	Average recovery (%)
Mango	Fruit	0.10	83.12	
		0.30	86.36	86.23
		0.50	89.23	
	Soil	0.10	81.14	
		0.30	84.23	84.67
		0.50	88.64	

*Mean of three replications

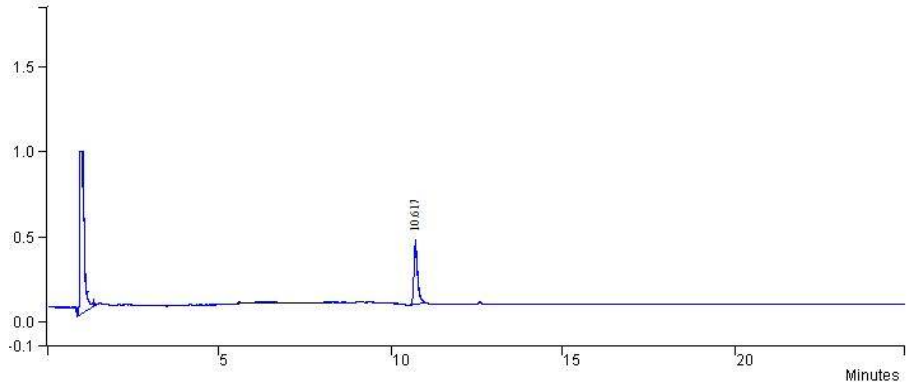
Table 2. Harvest time residues of thiamethoxam in mango

Treatment	Dose g a.i. ha^{-1}	Residues in µg g^{-1} (n=3)	
		Fruit	Soil
Thiamethoxam 25 WG	25	BDL	BDL
Thiamethoxam 25 WG	50	BDL	BDL
Untreated check	-	BDL	BDL

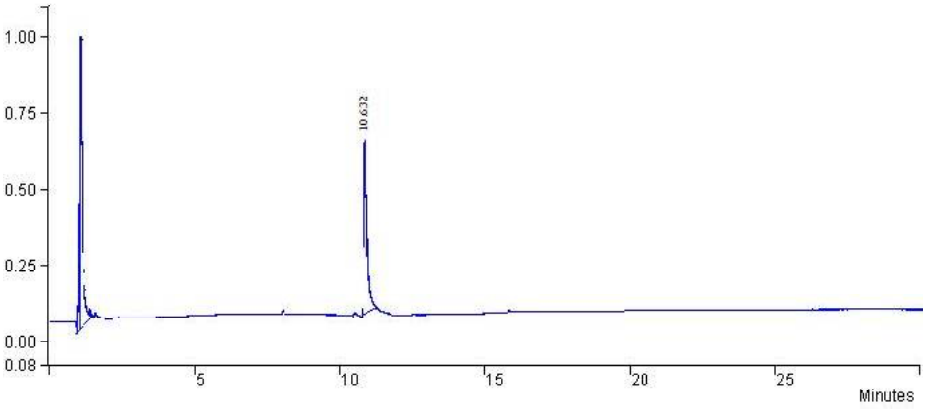
BDL – Below detectable limit (0.01 µg g^{-1})



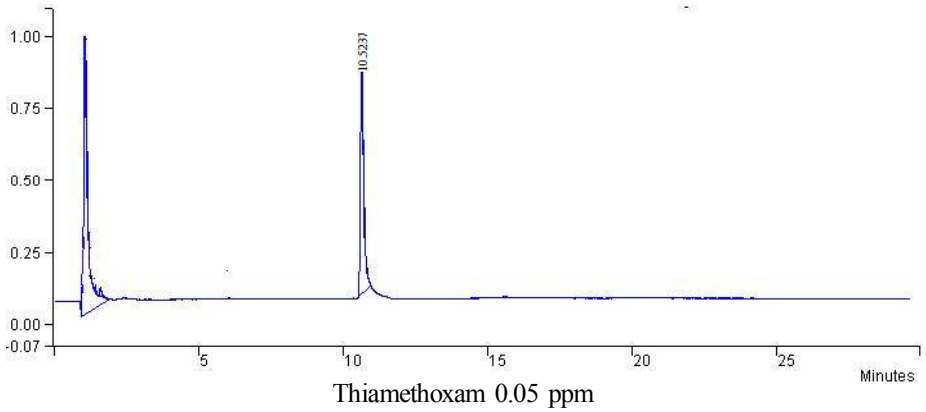
Thiamethoxam 0.003 ppm



Thiamethoxam 0.01 ppm



Thiamethoxam 0.03 ppm



Thiamethoxam 0.05 ppm

Fig.1 Standard chromatogram of thiamethoxam

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Received on 14-11-2017 Accepted on 19-11-2017