

Residue Dynamics of Imidacloprid and Hexaconazole on Mango

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Abstract

The experiment on residue dynamics of imidacloprid and hexaconazole on Mango was conducted during April-May, 2010 on mango variety Banganpalli. Three sprays of imidacloprid 17.8 SL @ 125 ml ha⁻¹ and 250 ml ha⁻¹ while hexaconazole 5 SL @ 100 ml ha⁻¹ and 200 ml ha⁻¹ were given at panicle emergence, marble stage and maturity stage. Initial deposits of imidacloprid at single and double dose treated samples were 0.52 and 0.83 ppm, respectively which dissipated to Below Detectable Level (BDL) at 5 and 7 days after last spray respectively. The initial deposits of hexaconazole at single and double dose treated samples were 1.82 and 2.16 ppm respectively dissipated to BDL at 10 and 15 days after second spray. The waiting periods for imidacloprid on mango at single and double dose was 4.27 and 5.19 days respectively, after last spray while the waiting period for hexaconazole on mango at single and double dose was 7.62 and 10 days respectively after last spray.

1. Introduction

Mango (*Mangifera indica* Linneaus), the king of fruits, is one of the oldest and choicest fruits of India for its high quality, palatability, tempting aroma and flavour. Andhra Pradesh being one of the major mango growing states is in forefront of mango fruit production and pulp industry. In India, apart from diseases, 250 insects and mites were recorded as pests in mango and to control these pests different insecticides and fungicides were sprayed at different stages from flower bud initiation to fruit maturity. Different insecticides and fungicides are used for the control of insect and disease problems in mango. The mango fruits will be consumed fresh and in order to avoid deleterious effects on human beings, the information on the time at which the mango fruits should be harvested after last spray is very much needed for safe consumption of mango fruits as raw. The information on safe harvest of mango fruits after insecticidal and fungicidal sprays is lacking. Keeping above facts in mind the present study was initiated.

2. Methods and Materials

The experiment on residue dynamics of imidacloprid and hexaconazole on Mango was conducted during April-May, 2010

on mango variety Banganpalli at AINP on Pesticide Residues, Rajendranagar, Hyderabad. Three sprays of imidacloprid 17.8 SL @ 125 ml ha⁻¹ and 250 ml ha⁻¹ while hexaconazole 5 SL @ 100 ml ha⁻¹ and 200 ml ha⁻¹ were given at panicle emergence, marble stage and maturity stage. The second spray was given one month after first spray. Third spray was given at the time of harvest. The Mango samples were collected for residue analysis of imidacloprid and hexaconazole at 0 (2hrs), 1, 3, 5, 7, 10 and 15 days after last spray. The samples were processed and residues of imidacloprid and hexaconazole were estimated under HPLC and GC-ECD respectively.

2.1. Extraction and cleanup of imidacloprid residues in mango pulp samples

Fifty grams of the mango pulp sample was placed in a 500 ml glass bottle with 250 ml acetonitrile and homogenized with high-speed blender for about 3 min by filtering through buchner funnel using fast filter paper overlaid with 10-15 g filter aid. Washed the blender jar and funnel with 100 ml acetonitrile, filtered and combined the acetonitrile fractions in 1,000 ml RB flask. Evaporated the acetonitrile by means of a rotary vacuum evaporator. Then 50 ml saturated sodium chloride solution

were added to aqueous remainder and transferred into a 500 ml separatory funnel, rinsed the RB flask with 100 ml n-hexane. After vigorous shaking, drained off the lower aqueous phase into 1,000 ml round-bottomed flask and discarded the organic phase.

The aqueous phase was returned into a separatory funnel and washed the RB flask with 100 ml n-hexane/ethyl acetate mixture (98:2)/(v/v). Shaken and drain the aqueous phase into a 250 ml separatory funnel. Discarded the organic phase. Then shaken the aqueous phase with 3 x 100 ml dichloromethane. Collected the lower dichloromethane phase in a 500 ml separatory funnel and washed with 50 ml of 0.01 molar aqueous potassium carbonate solution. Then discarded the aqueous phase. The dichloromethane phase was dried over anhydrous sodium-sulphate. Washed the sodium-sulphate with 50 ml of dichloromethane. Evaporated dichloromethane layer near to dryness. Washed the column with 10 ml ethyl acetate and then slurry packed with 4.5 g florisil (deactivated with 5% water) in 20 ml ethyl acetate. Then covered with 0.5 cm layer of anhydrous sodium sulphate. Allowed the solvent drain down to sodium sulphate layer. Dissolved the dry residue with a small amount of ethyl acetate. Applied the solution on the top of the column by means of a pipette. Rinsed the column with 20 ml of ethyl acetate. Eluted the residue with 20 ml acetonitrile. Concentrated the eluate just to dryness and dissolved the residue in 3.0 ml acetonitrile for analysis.

2.2. Extraction and cleanup of hexaconazole residues in mango pulp

Fifty grams of the mango pulp sample was blended with 350 ml of acetone/water (65:35 V/V) for 2 minutes at high speed. Filtered the extract and transferred the sample extract to one litre separatory funnel and extracted the residues with 200ml mixture of hexane: dichloromethane (1:1 v/v) by vigorous shaking for one minute. Transferred the lower aqueous phase to another one litre separatory funnel. Dried the organic phase of the first separatory funnel by passing through approximately 1.5" sodium sulphate supported on pre washed cotton in 4" funnel. To the separatory funnel containing aqueous phase, 10 ml saturated sodium chloride solution was added and shaken vigorously for 30 seconds. To this, 100 ml dichloromethane was added and shaken vigorously and dried the lower organic phase while passing through the same sodium sulphate. Repeated the extraction once more with 100 ml dichloromethane and dried as above. Then Concentrated the extract by using vacuum rotary evaporator. Concentration step was repeated in hexane to remove all the traces of dichloromethane and dissolved the extract in 10 ml n-hexane for analysis and analysed on GC-ECD.

3. Results and Discussion

Recovery studies were conducted prior to sample analysis by

fortifying the control samples with 0.01 and 0.1 ppm levels of imidacloprid and hexaconazole. The recovery of imidacloprid was 87.12 and 89.38 per cent at 0.01 and 0.1 ppm levels respectively while the recovery of hexaconazole was 85.79 and 84.91 per cent at 0.01 and 0.1 ppm levels respectively. These results were in conformity with the findings of Reddy and Reddy (2011)

Dissipation of imidacloprid and hexaconazole in mango after three sprays were depicted in Table 1 and 2 respectively. Initial deposits of imidacloprid at single and double dose treated samples were 0.52 and 0.83 ppm respectively dissipated to Below Detectable Level (BDL) at 5 and 7 days after last spray respectively. Sahoo et al., 2011 reported similar results in okra where residues of imidacloprid dissipated to below limit of quantification (0.01 ppm) at 5 and 7 days. Mohapatra et al., (2012) reported that imidacloprid in mango persisted for 15 days after application and variation may be due to change in climatic situation. The initial deposits of hexaconazole at single and double dose treated samples were 1.82 and 2.16 respectively dissipated to BDL at 10 and 15 days after second spray the present results were in conformity with Liang et al (2012) who reported that residues of hexaconazole in tomato dissipated to below detectable level at 15 days after last application. The maximum residue limits as suggested by Codex Alimentarius (1998) for both imidacloprid and hexaconazole in Mango was 0.02 ppm. The residue data was subjected to regression analysis to calculate the waiting period

Table 1: Dissipation of Imidacloprid in mango after three sprays of application

Day after second spray	Imidacloprid @125 ml ha ⁻¹		imidacloprid @250 ml ha ⁻¹	
	Residues mg kg ⁻¹	Dissipated (%)	Residues mg kg ⁻¹	Dissipated (%)
0	0.52	--	0.83	--
1	0.23	55.77	0.49	40.96
3	0.05	90.39	0.18	78.31
5	BDL	BDL	0.03	96.38
7	BDL	BDL	BDL	BDL
10	BDL	BDL	BDL	BDL
15	BDL	BDL	BDL	BDL
Soil	BDL	BDL	BDL	BDL
MRL (mg kg ⁻¹)	0.02		0.02	
T tol (days)	4.27		5.19	
T 1/2 (days)	1.87		1.96	
Regression equation	Y=0.7892-0.0141X		Y=0.9382-0.0237X	

BDL : Below detectable level @ 0.01 mg kg⁻¹

Table 2: Dissipation of Hexaconazole in mango after three sprays of application

Day after second spray	Imidacloprid @125 ml ha ⁻¹		imidacloprid @250 ml ha ⁻¹	
	Residues mg kg ⁻¹	Dissipated (%)	Residues mg kg ⁻¹	Dissipated (%)
0	1.82	-	2.16	-
1	1.23	32.42	1.53	63.00
3	0.79	56.59	0.93	56.95
5	0.24	86.81	0.36	83.33
7	0.03	98.35	0.19	91.20
10	BDL	BDL	0.02	99.07
15	BDL	BDL	BDL	BDL
Soil	BDL	BDL	BDL	BDL
MRL (mg kg ⁻¹)	0.02		0.02	
T tol (days)	7.62		10.00	
T 1/2 (days)	3.18		3.59	
Regression equation	Y=1.6281-0.0729X		Y=1.9362-0.0938X	

BDL : Below detectable level @ 0.01 mg kg⁻¹

and half life of imidacloprid and hexaconazole in Mango (Gunther and Blinn 1955; Haskins 1961). The waiting periods for imidacloprid on mango at single and double dose was 4.27 and 5.19 days respectively, after last spray while the waiting period for hexaconazole on mango at single and double dose was 7.62 and 10 days respectively after last spray.

4. Conclusion

The waiting periods for imidacloprid on mango at single and double dose was 4.27 and 5.19 days respectively, after last

spray while the waiting period for hexaconazole on mango at single and double dose was 7.62 and 10 days respectively after last spray.

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