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RESEARCH **P**APER

Residues and dissipation of lambda-cyhalothrin in pomegranate fruits

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Field and laboratory studies on residues and dissipation of lambda-cyhalothrin in pomegranate fruits were coducted during 2010 at the Pesticide Residue Analysis Laboratory, Department of Entomology, Mahatma Phule Krishi Vidyapeeth, Rahuri, Ahmednagar M.S. The studies revealed that residues of imidacloprid persisted up to 3 days and 5 days in arils, 5 days and 7 days in whole fruits and 7 days and 10 days in peel of pomegranate fruits at recommended and higher doses, respectively.

Key words : Pomegranate, Lambda-cyhalothrin, Residues

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INTRODUCTION

Pomegranate (*Punica granatum* L.) is a native of Iran and one of the favourite table fruits of tropical and sub-tropical regions. India ranks first in area (0.12 million ha) and production (9.0 million tonnes) of pomegranate followed by Iran with an area of 0.065 million ha and production 8.00 million tonnes. In India, Maharashtra ranks first (0.096 million ha) contributing 70 per cent of the total area under pomegranate followed by Karnataka (0.013 million ha) and Andha Pradesh (0.0051 million ha). Again average productivity of pomegranate in Maharashtra is very less *i.e* only 6.2 t ha^{-1}. In Maharashtra, Nashik district has an area of 0.0354 million ha followed by Solapur 0.0310 million ha, Ahmednagar 0.00639 million ha and Sangli 0.00630 million ha. However, the productivity of this crop in India is only 7.4 t ha^{-1} which is significantly lower than other pomegranate growing countries like Spain (18.5 t ha^{-1}), USA (18.3 t ha^{-1}) and Iran (9.23 t ha^{-1}) (Anonymous, 2008).

The insect pests and diseases play significant role in reducing the productivity of the crop. The pomegranate crop (*Punica granatum* L.) suffers from the attack of several insect and non-insect pests. Eighty six species of insect pests infecting pomegranate have been reported from various parts of the world (Zirpe, 1966). Thrips, *Scirtothrips dorsalis* (H) contributes major losses in pomegranate cultivation, both qualitatively and quantitatively. In order to protect the crop from pest problems; farmers are spraying a number of chemical pesticides on this crop. The disadvantages of pesticides as known as 4R (Resistance, Resurgence, Risk and Residue) are well known. Since this fruit is mostly accepted as a table purpose fresh fruit, pesticide residues in this crop are of very much concern. Pesticide residues are also becoming a major obstacle in reducing India's export to foreign market. The export scenario shows that India contributes only 5 per cent of International market while Spain is most dominant with 80 per cent share. Keeping in view the above facts and figures the present investigation was conducted to study the residues and dissipation of lambda-cyhalothrin in pomegranate fruits were carried out in the field experiment conducted in

Ambia bahar (*Summer* season) of 2010. Pesticide residues were analyzed in peel, arils and whole fruits separately, collected periodically after the third spray to decide the safety of treatments to consumers.

RESEARCH METHODOLOGY

The field experiments on bioefficacy of reduced risk insecticides against thrips, *Scirtothrips dorsalis* (H) of pomegranate were conducted during the *Ambia* bahar (*Summer*) and *Mrig* bahar (*Kharif*) seasons of 2010 on a five year old orchard of '*Bhagva*' variety at the Research Project on Arid Zone Fruits, Horticulture Farm, Department of Horticulture, Mahatma Phule Krishi Vidyapeeth, Rahuri, dist. Ahmednagar. Over all three sprays were given at an interval of 15 days, initiating first spray at the time of fruit setting. According to residue studies protocol prescribed by Central Insecticidal Board (CIB) two doses recommended (12.5 g a.i ha^{-i}) and double the recommended dose (25 g a.i ha^{-i}) were evaluated for analysis of residues. The marketable quality fruits of pomegranate weighing 1 kg were collected separately from each plot and packed properly in labeled polythene bags with rubber band and shifted to laboratory. Samples were collected at an interval of 0 (~ 2 hr), 1, 3, 5, 7, 10, 14 and 21 days after last spray for residue analysis. From composite samples by quartering method after cutting, 50 g representative samples were taken for extraction, cleanup and estimation as described under each compound. The analytical procedure followed for lambda-cyhalothrin is as follows.

Extraction :

Accurately weighed 50 g sample was blended in a high speed blender for extraction. Added 150 ml acetone and extracted for 30 minutes using a mechanical shaker. The extract was vacuum-filtered on Buchner funnel and re-extracted the cake with 2 x 75 ml of acetone. Acetone solvent in the combined extract was evaporated and added 50 ml saturated sodium chloride solution to the remaining aqueous layer. The aqueous extract was further cleaned up by partitioning.

Cleanup :

The aqueous extract was transferred quantitatively to a 1 lit. separating funnel with 50 ml distilled water and 100 ml hexane. The layers were partitioned by shaking vigorously and the aqueous layer was drained into another separating funnel. The aqueous layer was re-extracted by partitioning again with 2 x 50 ml hexane. The hexane phase was collected, combined and concentrated on RVE to approximately 10 ml.

A sintered disc glass column of 400 x 18 mm diameter was filled up with 10 g de-activated florisil overlaid with 15 mm layer of anhydrous sodium sulphate. The column was conditioned with 50 ml hexane and the concentrated sample extract was loaded onto the column. The sample flask was rinsed with 5 ml hexane and added to the column. The column was washed with 10 ml of the same solvent. The lambda-cyhalothrin residue was eluted with 150 ml ethyl acetate + hexane (5:95). The collected eluate was evaporated to near dryness and reconstituted in 10 ml ethyl acetate for GC-ECD analysis.

Estimation :

GC parameters :

Name of the instrument: Shimadzu Gas Chromatograph Model GC-2010 equiped with AOC-20 Auto injector and GC solution data software.

Gas chromatographic conditions :

Detector used : Electron capture detector (ECD)
Column used : DB-1, 30 m × 0.25 m × 0.25 µm
Temperature parameters : Injector : 250 ^{o}C
Detector : 300 ^{o}C
Oven temperature : 205^{o}C, 2 min hold, @ 2^{0}C/min 230^{0}C, 5^{o}C/min 245^{o}C 2 min hold.
Carrier gas : Nitrogen
Gas flow rate : 2.50 ml/min
Purge rate : 3.0 ml/min
Make up : 30 ml/min
Volume injected : 1 µl
Retention time : 15.71 min.

Calculation :

Calibration curve was prepared for imidacloprid by plotting the concentration of the calibration standards on X-axis and resulting peak height or area on Y-axis. Using regression analysis, the equation for the calibration curve was determined with respect to the X-axis. The concentration (C) of the analyte in the final solution was calculated from the measured peak height or area response (PR) and the least square co-efficient for the slope (m) and Y-axis intercept (b) as follows :

$$C = \frac{(PR - b)}{m}$$

The concentration (ug g^{-1}) of the analyte in the sample was calculated from the concentration (C) in final volume (V), the weight (W) of the sample that was extracted and the aliquot factor (AF) using the following equation :

$$\mu g\,g^{-1} \text{ or } ug\,g^{-1} = \frac{(C \times AF \times V)}{W}$$

The aliquot factor was calculated from the appropriate extraction and aliquot volumes for each sample type :

$$AF = \frac{\text{Total extraction volume}}{\text{Aliquot volume}}$$

The calibration of standards at various ng/ml levels showed good correlation between the concentration (X) and peak-height (Y) with ethe co-efficient of determination (r^2) averaging 0.955 or above.

RESEARCH FINDINGS AND ANALYSIS

The findings of the present study as well as relevant discussion have been presented in Table 1 and Fig. 1, 2 and 3.

Residues and dissipation of lambda-cyhalothrin in pomegranate fruits :

Arils :

Residues of lambda-cyhalothrin were detected by GC-ECD with the detectability level of 0.01 ppm. The initial deposit of 0.029 mg kg^{-1} in case of recommended dose of lambda-cyhalothrin (12.5 g a.i. ha^{-1}) reached below detection limit in 5 DAS in arils of pomegranate

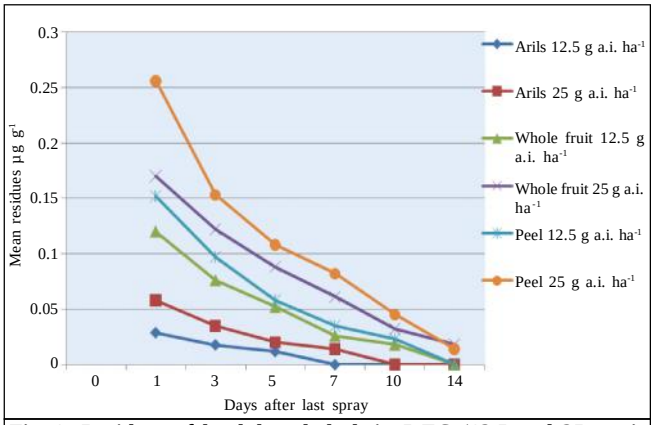


Fig. 1 : Residues of lambda-cyhalothrin 5 EC (12.5 and 25 g a.i. ha^{-1}) in pomegranate fruits

(Table 1). The dissipation of lambda-cyhalothrin in arils was in first order kinetics and the initial deposit of 0.029 mg kg^{-1} dissipated to 0.012 mg kg^{-1} at 3 DAS (Bonafos *et al.*, 2007; Seenivasan and Muraleedharan, 2009 and Caves and Gozukara, 2003). The estimated half-life value was 2.47 days and time taken to reach residue below detection limit of 0.01 ppm was 3.51 days at normal dose of 12.5 g a.i. ha^{-1}. At higher dose the initial deposit was 0.058 mg kg^{-1}at 0 DAS that dissipated to 0.035 (1 DAS), 0.020 (3 DAS) and 0.014 (5 DAS) mg kg^{-1}, respectively. The estimated half-life was 2.50 days and time required to reach below detection limit was 5.91 days (Ahrie *et al.*, 2008; Khey *et al.*, 2008; Campbell *et al.*, 2005, Pandey *et al.*, 2009 and Singh *et al.*, 2004).

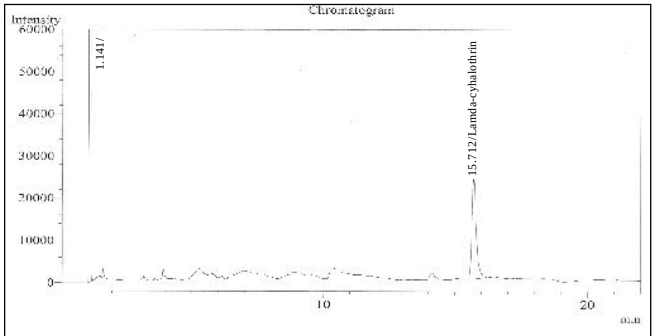
Whole fruit :

The whole fruit of pomegranate showed initial residue of 0.120 mg kg^{-1} (0 DAS) at 12.5 g a.i. ha^{-1}.

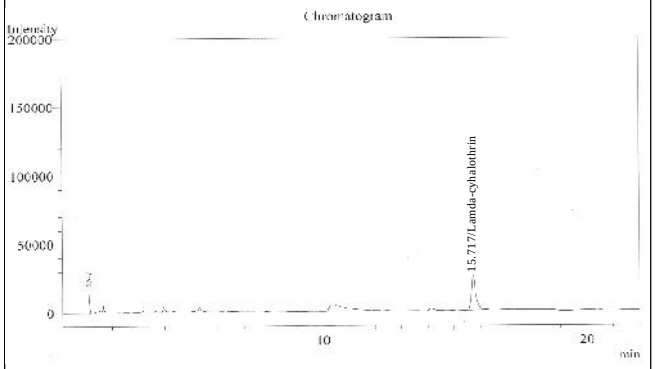
Table 1 : Residues of lambda-cyhalothrin in pomegranate fruits

Days after sprays	Lambda-cyhalothrin residue (mg kg^{-1})					
	12.5 g a.i. ha^{-1}	25 g a.i. ha^{-1}	12.5 g a.i. ha^{-1}	25 g a.i. ha^{-1}	12.5 g a.i. ha^{-1}	25 g a.i. ha^{-1}
	Arils		Whole fruit		Peel	
0	0.029	0.058	0.120	0.170	0.152	0.256
1	0.018	0.035	0.076	0.122	0.097	0.153
3	0.012	0.020	0.052	0.088	0.058	0.108
5	BDL	0.014	0.026	0.061	0.035	0.082
7	BDL	BDL	0.018	0.032	0.023	0.045
10	BDL	BDL	BDL	0.018	BDL	0.014
14	BDL	BDL	BDL	BDL	BDL	BDL
RL_{50} (Days)	2.47	2.51	2.59	3.11	2.63	2.60
T_{BDL} (Days)	3.51	5.91	8.95	12.62	9.91	12.03
T_{MRL} (Days)	1.04	3.40	6.36	9.51	7.28	9.43

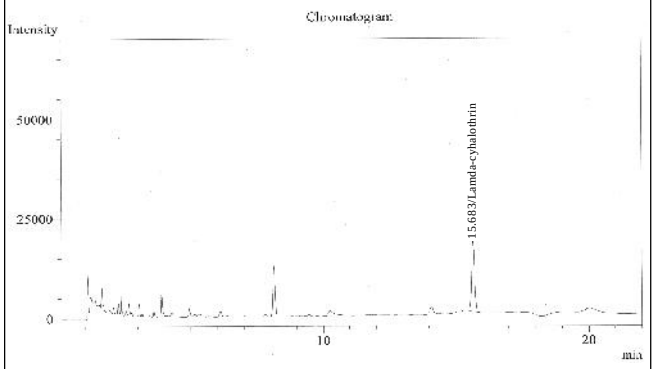
LOQ : 0.01 µg g^{-1} BDL : Below detection limit MRL : Maximum residue limit : 0.02 µg g^{-1}



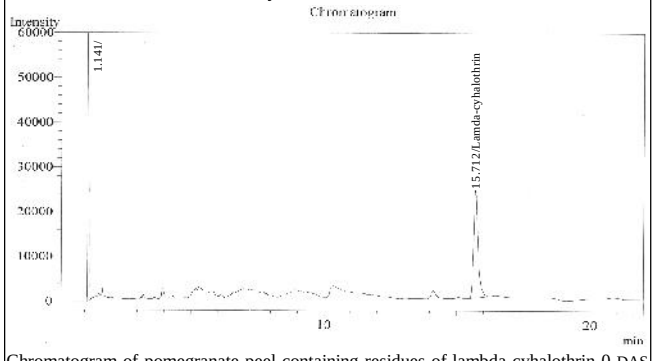
Typical chromatogram of lambda-cyhalothrin standard 0.01 µg g^{-1}



Chromatogram of pomegranate arils containing residues of lambda-cyhalothrin 0 DAS

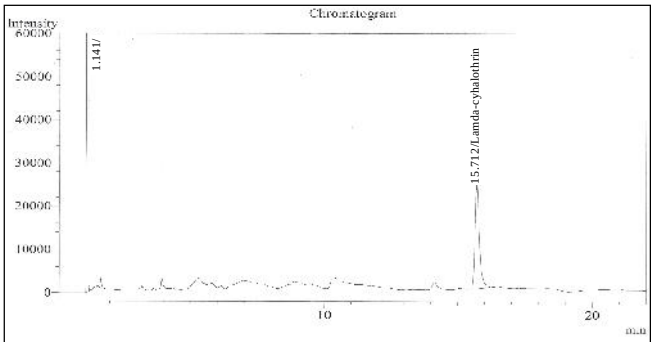


Chromatogram of pomegranate whole fruit containing residues of lambda-cyhalothrin 0 DAS

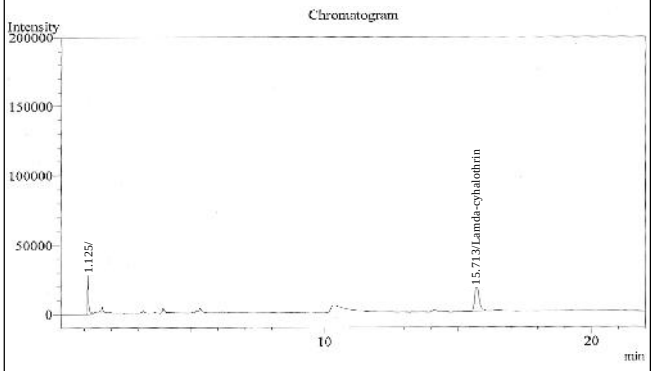


Chromatogram of pomegranate peel containing residues of lambda-cyhalothrin 0 DAS

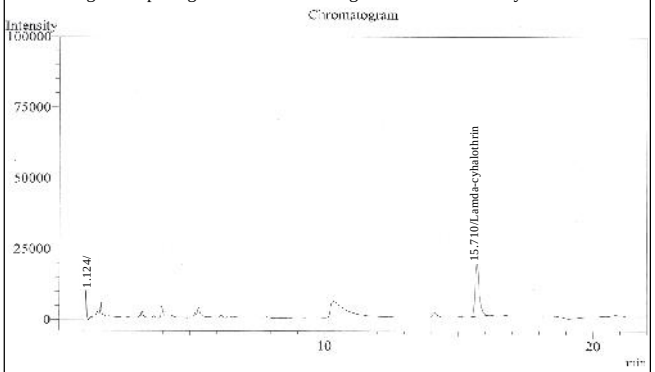
Fig. 2 : Typical chromatoram of lambda-cyhalothrin standard 0.02 µg g^{-1} and its residues in pomegranate fruits at 0 DAS



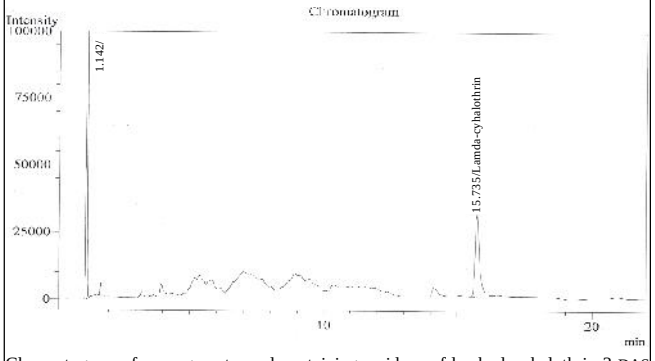
Typical chromatogram of lambda-cyhalothrin standard 0.01 µg g^{-1}



Chromatogram of pomegranate arils containing residues of lambda-cyhalothrin 3 DAS



Chromatogram of pomegranate whole fruit containing residues of lambda-cyhalothrin 3 DAS



Chromatogram of pomegranate peel containing residues of lambadacyhalothrin 3 DAS

Fig. 3 : Typical chromatogram of lambda-cyhalothrin standard 0.02 µg g^{-1} and its residues in pomegranate fruits at 3 DAS

At double dose of 25 g a.i. ha^{-1} the residues of lambda-cyhalothrin were 0.170 mg kg^{-1}at 0 DAS, 0.122 mg kg^{-1} at 1 DAS, 0.088 mg kg^{-1} at 3 DAS, 0.061 mg kg^{-1} 5 DAS, 0.032 mg kg^{-1} 7 DAS and 0.018 mg kg^{-1} at 10 DAS. The calculated half-lives for recommended and double the recommended dose were 2.59 to 3.11 days.

Peel :

The peel of pomegranate showed higher levels of residues at both the doses. At normal dose of lambda-cyhalothrin (12.5 g a.i. ha^{-1}) initial residues of 0.152 mg kg^{-1} degraded to 0.097 (1DAS), 0.058 (3 DAS), 0.035 (5 DAS) and 0.023 (7 DAS) mg kg^{-1} within 7 days. At double dose of 25 g a.i. ha^{-1} the initial residues of lambda-cyhalothrin were 0.256 mg kg^{-1} that dissipated to 0.014 mg kg^{-1} in 10 days. The half-life was found in the range of 2.63 days at normal dose and 2.60 days for double dose.

In absence of data on pomegranate, there is a limitation on discussion on present findings. Residues of lambda-cyhalothrin (35 g a.i. ha^{-1}) on brinjal fruits were studied by Dixit *et al*. (2001). They found that residues were below detectable limit after 7 days and the half-life of lambda-cyhalothrin on brinjal fruits was 1.45 to 2.54 days. The residues of lambda-cyhalothrin persisted in tomato fruits up to 5 days at 7.5 g a.i. ha^{-1} and 7 days at 15 and 30 g a.i. ha^{-1} (Mathirajan, 2002). Sharma *et al*. (2002) studied residues of lambda-cyhalothrin on cauliflower and reported that initial residues (0.81 to 1.59 mg kg^{-1}) dissipated quickly to reach below detectable limits within 10 to 15 days with the half-life (RL_{50}) of 2.2 to 2.4 days. Sureshkumar *et al* (2002) reported that residues of lambda-cyhalothrin (12.5 and 25g a.i. ha^{-1}) were below detectable levels in rice, bran, husk and straw at harvest. Banerjee *et al*. (2006) reported that residues of lambda-cyhalothrin at 25 and 50 g a.i. ha^{-1} were lost in grapes with a pre-harvest interval of 12.0 to 12.5 and 15.0 to 15.5 days, respectively. Similar work related with the present investigation was also conducted by Jadhav, 2006 on grapes.

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