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**Sakshi H Thantharate**
Department of Agril, Entomology, College of Agriculture, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dist. Ratnagiri, Maharashtra, India

**SD Desai**
Assistant Professor, Department of Agril. Entomology, College of Agriculture, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli, Ratnagiri, Maharashtra, India

**VS Desai**
Entomologist, Regional Fruit Research Station, Vengurla, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dist. Ratnagiri, Maharashtra, India

**AD Rane**
Associate Professor, College of Forestry, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dist. Ratnagiri, Maharashtra, India

**NK Hatwar**
Department of Agril, Entomology, College of Agriculture, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dist. Ratnagiri, Maharashtra, India

**YR Parulekar**
Assistant Professor, Department of Horticulture, College of Agriculture, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dist. Ratnagiri, Maharashtra, India

**Corresponding Author:**
**Sakshi H Thantharate**
Department of Agril, Entomology, College of Agriculture, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dist. Ratnagiri, Maharashtra, India

# Determination of residues of deltamethrin in Alphonso mango by HPLC

**Sakshi H Thantharate, SD Desai, VS Desai, AD Rane, NK Hatwar and YR Parulekar**

**Abstract**
Residues of deltamethrin, a pyrethroid insecticide has been determined in Alphonso mango by high - performance liquid chromatography with UV detection at 233 nm after extraction by chloroform and a silica gel clean-up. Deltamethrin was sprayed at recommended (9 ml/10 lit) and double recommended (18 ml/10 lit) doses on Mango trees. Mangoes from such treated trees were harvested and analyzed at 1, 5, 10, 15 and 25$^{th}$ day after spraying. Recommended dose showed residue of 0.14 ppm which was reduced to 0.08 ppm on 5$^{th}$ day and thereafter was not detectable. Double recommended dose recorded residue of 0.20 ppm which was reduced to 0.12 ppm on 5$^{th}$ day and later was not detectable.

**Keywords:** Deltamethrin, residue, Alphonso, HPLC

## Introduction
Mango (*Mangifera indica* L.) is appropriately called as "King of fruits" and also considered as "National fruit of India". The mango is a fleshy stone fruit belonging to the genus *Mangifera* from family Anacardiaceae. It is native to south Asia, from where it has been distributed worldwide to become one of the most cultivated fruit in the tropics. Its fame is mainly due to its excellent flavor, delicious taste and high nutritive value and became the choicest fruit of sub-continents. Native to southern Asia, especially eastern India, Burma, and the Andaman Islands, the mango has been cultivated, praised and even revered in its homeland since ancient time, where it has been cultivated for the last four thousand years. Maharashtra ranks 10$^{th}$ in production of mango which occupies an area of 166.76 thousand ha with the production of 791.36 metric tons in the year 2017-18. In Maharashtra, Alphonso, Kesar, Banganpalli, Dashehari, Amrapali, Rajapuri, Neelam, Totapuri and Langra are leading varieties of mango which are grown commercially. The Konkan region is famous and well known for mango production with an area of about 111.715 thousand ha with the production of 353.066 metric tons and productivity of 3.16 metric tons ha$^{-1}$ (Salvi *et al.*, 2018) [3].
The injudicious and indiscriminate application of insecticides to crops result in residues in food and food commodities with consequential hazards. Since most insecticides are toxic in nature, their continuous ingestion by human even in trace amounts, can result in accumulation in body tissues with serious adverse effects on health (Handa *et al.*, 1999) [2].
Many insecticides being highly stable continue to kill insect, for long period after their application. This ability of insecticide is called as "residue" which is both advantageous and disadvantageous. Advantageous because a single application achieve more kill, leave over a longer period covering even those insects which may not have been there at the time of application and disadvantageous because along with the harmful insects, the beneficial insects like parasitoids, predators and pollinators also run the risk of being killed due to prolonged residual action of toxicants (Shrivastava, 1988) [5]. The disadvantages of insecticide use are known as 4Rs (Resistance, Resurgence, Risk and Residue), are well known. Insecticide residues are also becoming a major obstacle in reducing India's export to international market. Among different varieties of mango, Alphonso variety of Konkan region is not only being famous in Maharashtra but also gaining more importance in many countries in view of export potential. Mango growers are therefore more worried about the damage inflicted by insect pests. To minimize the economic losses caused by these noxious pests, various pesticides are being used over the mango crop on massive scale. Due to lack of awareness, the farmers of our country do not follow the prescribed dosage and use pesticides at any stage of crop which results in accumulation of residue in fruits.
For the management of mango hoppers, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli has recommended a schedule of six sprays.

Though recommended schedule consist of six sprays, the farmers of Konkan region are taking indiscriminate sprays of insecticides to get higher yield. But the overdoses of insecticides make the residue problem, which might poison the fruits and are harmful for human health. Therefore, pesticide residue is becoming a major food safety concern of consumers and government.

The delicious Alphonso mango fruit is highly appreciated in many countries. Thus it opens tremendous opportunities for its export and fetch premium price in the world markets. Today market demands not only quality agricultural produce but also the safe and environment friendly production. To cope with the contemporary international market there is need to carry out systemic research on pesticide residue in mango fruits. In view of the importance of Mango and hazards caused by the insecticide residue, the research experiment was conducted for estimation of residues of deltamethrin in Alphonso mango by HPLC.

## Materials and methods

HPLC, rotary vacuum evaporator, mixer, buchner for vacuum filtration, homogenizer, weighing balance and Whatman filter paper no. 3.

## Reagents

Methanol and acetonitrile (HPLC grade); chloroform, hexane, dichloromethane, anhydrous sodium sulphate and silica gel (70 to 230 mesh A.S.T.M.) activated at 130°C during 10 hours (pesticide grade) were obtained from Merck (Darmstadt, Germany), double distilled water. Deltamethrin pesticide reference standard (>98 % of purity) was gracefully provided by Merck.

## Extraction of deltamethrin residues

Twenty-five grams of mango sample was cut and crushed in small pieces (according to the nature of the food) and transferred in a beaker (150 ml) with anhydrous sodium sulphate (20g) and chloroform (100 ml). The mixture was homogenized by a simple agitation for 5 minutes. The solvent was decanted on a Buchner funnel under suction using a Whatman filter paper no. 3. The operation was repeated with 50 ml of chloroform. The beaker was washed with 2 portions of 15 ml of chloroform, the washings was used to rinse the residue in the Buchner funnel. The filtrate was transferred in a separating funnel of 500 ml and the flask of the Buchner was washed with 2 portions of 50 ml of double distilled water which were added to the separating funnel. The aqueous phase was discarded and the chloroformic phase was dried by passing it through 15 g of anhydrous sodium sulphate in a glass column (of 300 mm length and 10 mm diameter). The chloroform extract was then concentrated to 5 ml by using a rotary vacuum evaporator under reduced pressure with the water-bath at 40°C.

## Clean up

By using a Pasteur pipette, the chloroform extract was transferred on to 5 g of silica gel column that has been activated at 130°C during 10 hours and slurry packed in hexane. The solution was let to pass. The flask of the rotary vacuum evaporator was rinsed with two portions of 10 ml of hexane which were also transferred in the column and let to pass; the elute was discarded and the process was repeated with 25 ml of hexane - dichloromethane (4+1), which was also discarded. Finally the column was eluted with 60 ml of dichloromethane which was collected. The dichloromethane was removed using the rotary vacuum evaporator at 40°C. The dry residue is dissolved with 10 ml of acetonitrile (or methanol or hexane). It was then ready for analysis.

## Analytical method

A suitable volume (20µl) of the extract was injected into the column and eluted with a mobile phase: acetonitrile - water (92:8) V/V at a flow rate of 0.85 ml/min. For detection, an absorption wavelength of 233 nm was employed.

## Results and Discussion

The results revealed that after one day, recommended dose of deltamethrin (9 ml/10 lit) showed residue of 0.14 ppm which was reduced to 0.08 ppm on 5$^{th}$ day and thereafter was not detectable at 10, 15 and on 25$^{th}$ day (Table 1). This indicated that the residue of deltamethrin at recommended dose of 9 ml/10 litre lasts up to 5 days and thereafter it is not detectable.

**Table 1:** Residue of Deltamethrin in mango fruits at periodic interval

| Dose ml/10 lit | Average residue (ppm) at periodic interval (days) | | | | |
|---|---|---|---|---|---|
| | 1 | 5 | 10 | 15 | 25 |
| 9 | 0.14 | 0.08 | ND | ND | ND |
| 18 | 0.20 | 0.12 | ND | ND | ND |
| SE ± | 0.02 | 0.03 | ND | ND | ND |
| CD at 1 % | 0.05 | - | - | - | - |
| Significance | Sig. | NS | - | - | - |

ND: Not Detectable

The results of double recommended dose of deltamethrin (18 ml/10 lit.) recorded residue of 0.20 ppm after one day which was reduced to 0.12 ppm on 5$^{th}$ day and thereafter was not detectable at 10, 15 and on 25$^{th}$ day. This indicated that the residue of deltamethrin at double recommended dose of 18 ml/10 litre lasts up to 5 days and thereafter it is not detectable. Data on residue of deltamethrin at one day after spraying revealed that the recommended dose 9 ml/10 litres of water was found to be significantly superior over double dose of deltamethrin i.e. 18ml/10litre. Whereas, at 5 days after spraying the residue at both the doses was non-significant.

The results obtained by Sen and Chowdhury (1999) [4] are in conformity with the results of the present investigation. They reported no residue of deltamethrin in brinjal after 10 days of application. Dissipation pattern of deltamethrin in mango fruits was also studied in the present investigation. Data pertaining to dissipation study are presented in Table 2.

**Table 2:** Dissipation pattern of Deltamethrin in mango fruits

| Dose ml/10 lit | Percent loss of residue at periodic interval (days) | | | | | Half-life (days) | Waiting period (days) |
|---|---|---|---|---|---|---|---|
| | 1 | 5 | 10 | 15 | 25 | | |
| 9 | 0.00 | 57.14 | 100.00 | - | - | 4.35 | 6.52 |
| 18 | 0.00 | 40.00 | 100.00 | - | - | 6.02 | 9.25 |
| SE ± | - | 2.25 | - | - | - | 0.85 | 1.28 |
| CD at 1 % | - | 5.50 | - | - | - | 1.94 | 2.50 |
| Significance | - | Sig. | | | | | |

Per cent loss of Deltamethrin residue at a dose of 9 ml/10 litre on 5$^{th}$ day was 57.14 which was totally lost at 10 days. The half-life of Deltamethrin at a dose of 9 ml per 10 litre was observed to be 4.35 days and waiting period was 6.52 days. The insecticide Deltamethrin at a dose of 18 ml per 10 litre dissipates 40 per cent on 5$^{th}$ day and it was totally lost on 10$^{th}$ day. The half-life of Deltamethrin at a dose of 18 ml per 10 litre was recorded to be 6.02 days and waiting period was 9.25 days. This indicated that the higher dose of Deltamethrin

dissipates faster than recommended dose. Similar kind of results have been reported in earlier findings in Mango (Awasthi, 1988) [1] and brinjal (Sen and Chowdhury, 1999) [4].

## Conclusion

The method of extraction – clean up that we have adapted and tested that consists of an extraction with chloroform followed by clean up on activated silica gel appears to be completely satisfactory.

From the results of the present investigation it can be concluded that the insecticide deltamethrin does not leave residue for a longer period therefore can be used at later stage of the crop.

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