

**ESTIMATION OF INSECTICIDAL RESIDUE IN  
ALPHONSO MANGO**

**By**

**Ms. Thantharate Sakshi Haresh**

**B.Sc. (Ag.)**

**DEPARTMENT OF AGRICULTURAL ENTOMOLOGY,  
FACULTY OF AGRICULTURE,  
DR. BALASAHEB SAWANT KONKAN KRISHI VIDYAPEETH,  
DAPOLI 415 712, DIST. RATNAGIRI  
MAHARASHTRA, INDIA**

**AUGUST, 2020**

# **ESTIMATION OF INSECTICIDAL RESIDUE IN ALPHONSO MANGO**

A thesis submitted to the

**DR. BALASAHEB SAWANT KONKAN KRISHI VIDYAPEETH, DAPOLI**  
(Agricultural University) Dist. Ratnagiri (MS)

*In partial fulfillment of the requirements for the award of the degree of*

## **MASTER OF SCIENCE**

### **(AGRICULTURE)**

In

### **AGRICULTURAL ENTOMOLOGY**

By

**Ms. THANTHARATE SAKSHI HARESH**

**B.Sc. (Ag.)**

**DEPARTMENT OF AGRICULTURAL ENTOMOLOGY,  
FACULTY OF AGRICULTURE,  
DR. BALASAHEB SAWANT KONKAN KRISHI VIDYAPEETH,  
DAPOLI 415 712, DIST. RATNAGIRI  
MAHARASHTRA, INDIA**

**AUGUST, 2020**

# **ESTIMATION OF INSECTICIDAL RESIDUE IN ALPHONSO MANGO**

A thesis submitted to the

**FACULTY OF AGRICULTURE**  
**DR. BALASAHEB SAWANT KONKAN KRISHI VIDYAPEETH, DAPOLI**  
(Agricultural University)  
**Dist. Ratnagiri (MS)**

*In partial fulfilment of the requirements for the degree of*

## **MASTER OF SCIENCE (AGRICULTURE)**

**In**

### **AGRICULTURAL ENTOMOLOGY**

**By**

**Ms. Thantharate Sakshi Haresh**  
**B.Sc. (Ag.)**

**Approved by the Advisory Committee**  
**Chairman and Research Guide**

**(S. D. Desai)**  
Assistant professor,  
Department of Agril. Entomology,  
College of Agriculture, Dapoli

**Members:**

**(V. S. Desai)**  
**Entomologist,**  
**Regional Fruit Research Station,**  
**Vengurla**

**(Y. R. Parulekar)**  
Assistant Professor,  
Department of Horticulture,  
College of Agriculture, Dapoli

**(A. D. Rane)**  
Associate Professor,  
College of Forestry, Dapoli,  
Dr. B.S.K.K.V, Dapoli

**Dr. S.D. Desai**

**M.Sc. (Agri.), Ph.D.**

Assistant professor,  
Department of Agril. Entomology,  
College of Agriculture, Dapoli  
Dist. Ratnagiri, Maharashtra  
Dr. B.S.K.K. V., Dapoli 415 712

**CERTIFICATE**

This is to certify that the thesis entitled, **“Estimation of insecticidal residue in alphonso mango”** submitted to the Faculty of Agriculture, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli, Dist. Ratnagiri, Maharashtra State, in the partial fulfilment of the requirements for the degree of **MASTER OF SCIENCE (Agriculture) in AGRICULTURAL ENTOMOLOGY**, embodies the results of a piece of bona-fide research work carried out by **THANTHARATE SAKSHI HARESH (Regd. No.: ADPM/18/2600)** under my guidance and supervision and that no part of this thesis has been submitted for any other degree or diploma or published in other form. All the assistance and help received during the course of investigation and the sources of literature have been duly acknowledged by her.

**Place :** Dapoli

**Date:**     /     /

**(S. D. Desai)**  
Chairman  
Advisory Committee  
and  
Research Guide

## **CONTENTS**

<b>CHAPTER</b>	<b>PARTICULARS</b>	<b>PAGE No.</b>
<b>I</b>	<b>INTRODUCTION</b>	<b>1-5</b>
<b>II</b>	<b>REVIEW OF LITERATURE</b>	<b>6-31</b>
<b>III</b>	<b>MATERIAL AND METHODS</b>	<b>32-41</b>
<b>IV</b>	<b>RESULTS AND DISCUSSION</b>	<b>42-55</b>
<b>V</b>	<b>SUMMARY AND CONCLUSION</b>	<b>56-60</b>
	<b>LITERATURE CITED</b>	<b>I-VIII</b>
	<b>APPENDIX</b>	<b>-</b>

## LIST OF TABLES

<b>Table No.</b>	<b>Particulars</b>	<b>Page No.</b>
1.	Percent mean recovery of pesticides in mango fruits at different levels of concentration on first day after spraying	43
2.	Residue of Deltamethrin in mango fruits at periodic interval	43
3.	Dissipation pattern of Deltamethrin in mango fruits	44
4.	Residue of Lambda cyhalothrin in mango fruits at periodic interval	45
5.	Dissipation pattern of Lambda cyhalothrin from Mango fruits	47
6.	Residue of Imidaclopridin Mango fruits at periodic interval	48
7.	Dissipation pattern of Imidacloprid from mango fruits	48
8.	Residue of Thiamethoxam in mango fruits at periodic interval	49
9.	Dissipation pattern of Thiamethoxam from mango fruits	51
10.	Residue of Dimethoate in mango fruits at periodic interval	52
11.	Dissipation pattern of Dimethoate from mango fruits	52
12.	Residue of different insecticides in mango fruits of farmer's field	54

## LIST OF PLATES

<b>Plate No.</b>	<b>Title</b>	<b>Between Page No.</b>
I	Application of pesticidal treatments	34-35
II	High Performance Liquid Chromatograph equipped with PDA	39-40
III	Extraction and clean-up steps for residue analysis	39-40

**DEPARTMENT OF AGRICULTURAL ENTOMOLOGY,  
COLLEGE OF AGRICULTURE, DAPOLI  
DR. BALASAHEB SAWANT KONKAN KRISHI VIDYAPEETH, DAPOLI,  
DIST. RATNAGIRI, MAHARASHTRA**

---

<b>Title of thesis</b>	: Estimation of insecticidal residue in Alphonso mango
<b>Name of Student</b>	: Ms. Thantharate Sakshi Haresh
<b>Registration Number</b>	: ADPM/18/2600
<b>Name &amp; designation of Research guide</b>	: Dr. S. D. Desai Assistant Professor Department of Agril. Entomology College of Agriculture, Dapoli
<b>Year of Submission</b>	: 2019-20

---

**ABSTRACT**

Estimation of insecticidal residue was taken up with objectives to estimate the residue of recommended insecticides in Alphonso mango and insecticidal residue from farmer orchards.

Data on residue of deltamethrin sprayed at recommended and double doses one day after spraying revealed that recommended dose of deltamethrin (9 ml/10 lit.) showed residue of 0.14 ppm which was reduced to 0.08 ppm on 5<sup>th</sup> day and thereafter was not detectable at 10, 15 and on 25<sup>th</sup> day. Deltamethrin sprayed at double than recommended dose (18ml/10 litre) showed residue of 0.20 ppm after one day which was reduced to 0.12 ppm on 5<sup>th</sup> day and thereafter was not detectable at 10, 15 and on 25<sup>th</sup> day.

Per cent loss of deltamethrin residue at recommended dose on 5<sup>th</sup> day was 57.14 which was totally lost at 10 days. The half-life of deltamethrin at a recommended dose was observed to be 4.35 days and waiting period was 6.52 days. Deltamethrin at a



double dose dissipates 40 per cent on 5<sup>th</sup> day and it was totally lost on 10<sup>th</sup> day. The half-life of deltamethrin at a double dose was recorded to be 6.02 days and waiting period was 9.25 days.

Data on residue of lambda cyhalothrin sprayed at recommended (6 ml/10 lit.) and double doses (12 ml/10 lit.) at 1,5,10,15 and 25 days after spraying revealed that recommended dose showed residue of 0.84 ppm which was reduced to 0.48, 0.21 and 0.04 ppm on 5, 10 and 15 days after spray, respectively. On 25<sup>th</sup> day the residue of lambda cyhalothrin was below detectable level.

The results of lambda cyhalothrin sprayed at double than recommended dose (12ml/10 lit.) revealed that after one day, double recommended dose of lambda cyhalothrin (12 ml/10 lit.) recorded residue of 1.40 ppm which was reduced to 0.95, 0.53 and 0.10 ppm on 5,10 and 15 days after spraying. The residue of lambda cyhalothrin at 25 days after spraying was below detectable level.

Per cent loss of lambda cyhalothrin residue at a dose of 6 ml/10 litre on 5<sup>th</sup> day was 42.83 per cent which was reduced to 75.00 and 95.23 per cent at 10 and 15 days after spraying. The residue of lambda cyhalothrin was totally lost at 25 days after spraying. The half-life of lambda cyhalothrin at a dose of 6 ml per 10 litre was observed to be 8.12 days and waiting period was 18 days. The insecticide lambda cyhalothrin at a dose of 12 ml per 10 litre dissipated 32.14 per cent on 5<sup>th</sup> day then it was reduced to 62.14 and 92.85 per cent at 10 and 15 days after spraying and later on it was totally lost on 25<sup>th</sup> day. The half-life of lambda cyhalothrin at a dose of 12 ml per 10 litre was recorded to be 8.75 days and waiting period was 19 days.

Imidacloprid residue of the dose 3 ml per 10 litre in HPLC on day one after spraying was 0.12 ppm which reduced to 0.06 ppm at 5 days after spraying. It was below detectable level on 10<sup>th</sup> day after spraying. The residue of imidacloprid at double than recommended dose of 6 ml per 10 litre at one day after spraying was 0.16 ppm which was reduced to the extent of 0.10 and 0.04 ppm at 5 and 10 days after spraying and later on it was not detectable.

Per cent loss of imidacloprid residue at a dose of 3 ml/10 litre on 5<sup>th</sup> day was 50.00 which was reduced to 100 per cent on 10<sup>th</sup> day after spraying. The half-life of imidacloprid at a dose of 3 ml per 10 litre of water was found to be 5 days and waiting period was 10 days. The insecticide imidacloprid at a dose of 6 ml per 10 litre dissipated 37.50 per cent on 5<sup>th</sup> day then it was reduced to 75.0 per cent at 10<sup>th</sup> day after spraying and later on it was totally lost on 15<sup>th</sup> day. The half-life of imidacloprid at a dose of 6 ml per 10 litre was recorded to be 7.5 days and waiting period was 15 days.

The residue of thiamethoxam at a recommended dose of 1 g per 10 litre of water at one day after spraying was 1.21 ppm which was reduced to 0.53, 0.25, 0.13 and 0.06 at 5,10,15 and 25 days respectively. The residue of thiamethoxam at double than recommended dose at 2 g per 10 litre of water at one day was 2.02 ppm which was reduced to the extent of 1.02, 0.42, 0.20 and 0.12 ppm at 5,10,15 and 25 days after spraying.

The insecticide thiamethoxam at a dose of 1 g per 10 litre was dissipated to the extent of 56.19 per cent at five days after spraying and then it was reduced to 79.33, 89.25 and 95.04 per cent at 10, 15 and 25 days respectively. Whereas, double than recommended dose of thiamethoxam was reduced from 49.50, 79.20, 90.09 and 94.05 per cent at 5,10,15 and 25 days after spraying respectively. The half-life of thiamethoxam at a recommended dose was 4.80

days and waiting period was 20 days. The half-life and waiting period of double than recommended dose of thiamethoxam was reported to be 5.20 and 20 days respectively.

The residue of dimethoate at a recommended dose of 10 ml per 10 litre of water at one day after spraying was 1.92 ppm which was reduced to 0.57 and 0.12 ppm on 5 and 10 days after spraying respectively. The residue of dimethoate was not detectable after 15 days. Whereas, the residue of dimethoate at double than recommended dose of 20 ml per 10 litre was 3.58 ppm at one day after spraying which was reduced to 1.04 and 0.19 ppm at 5 and 10 days respectively. The insecticide dimethoate was not detectable after 15 days after spraying in mango fruits.

The insecticide dimethoate at a dose of 10 ml per 10 litre of water was lost to the extent of 70.31 per cent on 5<sup>th</sup> day which was then lost to 94.16 and 100 per cent at 10 and 15 days after spraying. Whereas, at double than recommended dose of 20 ml per 10 litre of water was lost from 70.94, 94.69 and 100 per cent at 5, 10 and 15 days after spraying. The half-life of dimethoate at single and double dose was 3.68 and 3.69 days whereas, waiting period was 11 and 12 days respectively.

The mango fruits as per treatment were brought from farmers field for the estimation of insecticide residue. The residue of the insecticides deltamethrin, lambda cyhalothrin, imidacloprid, thiamethoxam and dimethoate was estimated by using HPLC at College of Forestry.

The data on residue of the five insecticides under study in mango fruits of the farmers filed are presented in Table 12. Data revealed that the residue of deltamethrin was not detectable. The residue of lambda cyhalothrin was 0.24 ppm which uses University recommended schedule. The residue of the insecticide lambda

cyhalothrin was 0.18, 0.44 and 0.48 ppm from the mango fruits of the farmersfield those who practice 5, 6-10 and more than 10 sprays respectively. The residue of imidacloprid from the mango fruits of the farmer's those use University recommended schedule was 0.02 ppm. The residue of imidacloprid was 0.38, 0.40 and 0.42 ppm those who use 5, 6-10 and more than 10 sprays respectively. The residue of thiamethoxam from the mango fruits of the farmers who use university schedule was 0.84 ppm. Whereas, the residue was 0.84, 0.18 and 1.24 ppm from the mango fruits of the farmers those practice up to 5 sprays, 6-10 sprays and more than 10 sprays. The residue of dimethoate from the mango fruits of the farmers field those practice University recommended schedule was 0.84 ppm. The farmers use up to 5 sprays, 6-10 sprays and more than 10 sprays recorded not detectable, 0.02 ppm and not detectable residue of dimethoate respectively.

## **ACKNOWLEDGEMENT**

*Determination and hard work leads to success but not without the guidance of a real critic. After successfully completing the post graduate educational journey, I look back and find that though mine has been a fairly sail, it has been memorizing extravaganza of memorable experiences. At this gratifying moment of completion of my research work, I feel obliged to record my gratitude to those who have helped me.*

*It gives me immense pleasure to express my deep sense of gratitude to my Research guide and Chairman of my Advisory committee Dr. S. D. Desai, Assistant Professor, Department of Agril. Entomology, College of Agriculture, Dapoli for his ardent concern, stirring guidance and assiduous assistance during the investigation and in the preparation of this thesis. His constant guidance and encouragement buoyed me all along and helped me in every respect in M. Sc. studies and research work. It was privilege that he was always available to solve the problem that would arise during the course of research and thesis writing. No words are ample to express my thanks to my research guide.*

*It gives me great pleasure to express my profound gratitude and heartfelt respect to my Advisory Committee members, Dr. V. S. Desai Entomologist, Regional Fruit Research Station, Vengurla is a person with an amicable and positive disposition, Sir has always made himself available to clarify my doubts despite his busy schedules and I consider it as a great opportunity to do my M. Sc. programme under his guidance and to learn from his research expertise. I also confess my respectful gratitude to Prof. Y. R. Parulekar, Assistant Professor, Department of Horticulture, College of Agriculture, Dapoli and Dr. A. D. Rane Associate Professor, College of*

*Forestry, Dapoli, for giving me valuable guidance and timely help during the course of my post graduation studies.*

*It is my proud privilege to record my deep sense of appreciation and sincere thanks to Dr. A. L. Narangalkar, Head, Department of Agril. Entomology, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth Dapoli for his valuable suggestions during the course of research work,*

*I extend my sincere thanks to Dr. (Mrs.) Kumud. V. Naik, Professor (CAS), Department of Agril. Entomology, Dr. S. K. Mehendale, Professor, Department of Agril. Entomology, Dr. B. D. Shinde, Assistant Professor, Department of Agril. Entomology, Dr. M. S. Karmarkar Assistant Professor, Department of Agril. Entomology, Dr. S. N. Kale, Assistant Professor, Department of Agril. Entomology, for their co-operation and guidance during this investigation.*

*I place on record my sincere thanks to Dr. S. D. Sawant, Hon. Vice-Chancellor, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli. Dr. S. S. Narkhede, Dean, Faculty of Agriculture, and Dr. U. V. Mahadkar, Associate Dean, College of Agriculture, Dapoli, for their valuable guidance and providing necessary facilities for conducting this study.*

*I am sincerely thankful to College of Horticulture, Dapoli for providing research plot and materials during my research work. I convey my special thanks to Ms. Purnima Arekar for her valuable guidance during my research work,*

*I convey my thanks to Mr. R. H. Mhatre, Agril. Assistant, Mr. Ambarish Sanas, Sanvi mam, Pagade kaka, Viju kaka, Dhamne kaka, Rale kaka and all staff members of my department who helped me throughout the period of my stay at Dapoli.*

*My vocabulary fails to get the words to express my respect and sense of gratitude towards my beloved family members, my father Mr. HareshNatthulalThantharate, my mother Mrs. JaishreeHareshThantharate, my beloved Brother Tanay, my aunt Ms. Nita Katakwar for their everlasting love, constant inspiration Prayer, support and guidance. My head bows respectfully before my all relatives who inspired me with love and affection.*

*On a long journey such as this, one encounters a number of fellow travellers moving towards the same destination. This companies and enthusiasm and zest to the journey making it enjoyable. I was fortunate to travel the path of knowledge with my classmates Pranjali, Nikhil, Akshay, Pratyay, Madhuri, Aishwarya and Huzaiifa. I wholeheartedly express my special thanks to my friends Ashish sir, Priyanka, Parag, Siddharth for their excellent company, inspiration, moral support, boost up and best friendship.*

*I am thankful to my seniors Chavan sir, Sagar sir, Priti mam, Dubale sir, Hemant sir and my beloved juniors Sanket and Sneha. My all Sr. M.Sc., Jr. M.Sc., Ph. D. friends for their enthusiastic company and cooperation who helped me directly or indirectly and offered their excellent company and warm affection throughout my stay in this University. Finally, I owe gratitude to all those whom, I might have forgotten.*

*Place : Dapoli*

*Date :    /    /2020*

*(Thantharate Sakshi Haresh)*

## CHAPTER I

### 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-continent. 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.

Number of varieties of mango having unusual diversity of flavor and taste are under cultivation in India. In many parts of the country mango serves as staple article of food for several months during the year. Out of total world production of mango, India's share is about 65 per cent (Mukharjee *et al.*, 2007). India is the major producer of mango in the world. In the year 2017-18, 2258.10 thousand ha area of India was covered with mango, with a production of **21822.32 metric** tons (Anonymous, 2018).

Mango is generally sweet, although the taste and texture of the flesh varies across cultivars, some having a soft, pulpy texture similar to an overripe plum. In India, mango is consumed mainly as ripe fruit which is also preserved in various forms, whereas, raw mango preserved and consumed as pickles, moramba, curries and chutneys of all sorts. The kernel is dried, roasted and eaten. During the scarcity period, the same is



ground into flour and eaten as gruel. The bark is used in tanning leather, while the timber is being utilized in various ways.

Besides these, mango fruit have a good nutritive value, enriched in  $\beta$ -carotene, vitamin A, vitamin B complex, vitamin C, minerals, digestible sugars and trace elements of anti-oxidants. Furthermore, every 100 g of mango fruit pulp contains 81.7 g water, 16.0 g carbohydrates, 0.7 g protein, 0.4 g fat and 0.1 g fibers, which are variable in different mango cultivars.

Maharashtra ranks 10<sup>th</sup> 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<sup>-1</sup> (Salviet *al.*, 2018).

Introduction of various Agri-export zones in India has made mango a good source of foreign exchange. Indian producers find it easier to expand sales to the European Union. Europe's acceptance of different varieties is greater, because of a large demand from Indian immigrant groups. Moreover, a lion's share of Indian mango goes to the Gulf countries. Now a day, the demands of mango fruits have been increased in many developed and developing countries in the form of canned or fresh fruits.

The crop is attacked by several insect pests and diseases, affecting both the quality and quantity of mango fruits. About 150 insect pests have been reported so far but hardly half a dozen are of major importance (Butani, 1974). Among them, mango hopper complex, *Idioscopusclypealis* L., *Idioscopusniveosparsus* L. and *Amritodusatkinsoni* L., fruit fly, *Bactrocera dorsalis* Hendel, mealy bug, *Drosicha mangiferae* Green, bark eating caterpillar, *Inderbela quadrinotata* Wlk, stem borer, *Batocera rufomaculata* DeGeer, and scale insect, *Aspidiotus destructor* Signoret are major one. Among the insect pests, the commonest and perhaps the most serious and destructive one is mango hopper complex (Shah *et al.*, 1973).

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).

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). 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 hopper, 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 entitled “Estimation of insecticidal residue in Alphonso mango” was conducted with the following objectives:

1. To estimate the residue of recommended insecticides in Alphonso mango
2. To estimate the insecticidal residue from farmers orchards

## **CHAPTER II**

### **REVIEW OF LITERATURE**

The present investigation was carried out in the Konkan region of Maharashtra to study the “Estimation of insecticidal residue in Alphonso mango” in the year 2020. The available literature pertaining to the present investigation have been reviewed and presented with respect to the objectives under the following headings.

- 2.1 To estimate the residue of recommended insecticides in Alphonso mango
- 2.2 To estimate the insecticidal residue from farmers orchards

#### **2.1 To estimate the residue of recommended insecticides in Alphonso mango**

##### **2.1.1 Deltamethrin**

Awasthi (1988) studied persistence of 4 synthetic pyrethroids on mango fruits. Permethrin 0.02 per cent, cypermethrin 0.01 per cent, and fenvalerate 0.01 per cent were found to persist for three weeks dissipating their residues at the respective half-life values of 5.67, 5.83 and 5.33 days to require correspondingly 7.27, 3.45 and 11.27 days waiting periods based on their tolerance limits. However, deltamethrin 0.002 per cent persisted only for one week to dissipate at the half-life of 3.81 days and require no waiting period. None of these insecticides penetrated into the pulp of fruit at any stage after spraying.

Hafeez and Rizvi (1993) studied residues of some synthetic pyrethroids and monocrotophos in/on okra fruits. The residues of deltamethrin (applied at 12 g a.i. ha<sup>-1</sup>), cypermethrin (60 g a.i. ha<sup>-1</sup>), permethrin (120 g a.i. ha<sup>-1</sup>), fenvalerate (120 g a.i. ha<sup>-1</sup>) and

monocrotophos (320 g a.i. ha<sup>-1</sup>) on green fruits of okra were 0.02 – 0.41 ppm on the 2<sup>nd</sup> day, and 0.01- 0.15 ppm on the 5<sup>th</sup> day after application. They reported 2 days waiting period for deltamethrin, cypermethrin and permethrin, whereas 5 days waiting period were sufficient for fenvalerate and monocrotophos.

Sen and Chowdhury (1999) conducted field experiment to study long term residue and persistence study of deltamethrin in brinjal. It has been revealed from the study that, about 92.98 per cent initial residue of deltamethrin disappeared in brinjal within 5 days at 10, 12.5 and 25 g a.i. ha<sup>-1</sup>. No residue has been detected in brinjal after 10 days of application. Half-life of insecticide varies within 1.03 to 1.15 days. Thus, a safe waiting period of 5 days is recommended for brinjal in all the three doses.

Kumari *et al.* (2002) determined the magnitude of pesticide contamination in six popularly grown summer vegetables (okra, smooth gourd, bitter gourd, cucumber, tomato and brinjal) in Haryana and found that all the 60 market samples of vegetables were found to be contaminated with 3 or 4 insecticides of each chemical groups i.e. organochlorine, organophosphorus, synthetic pyrethroid and carbamate. Okra fruits were contaminated with organochlorine (HCH, DDT, endosulfan and aldrin), synthetic pyrethroids (cypermethrin, deltamethrin and fenvalerate), organophosphate (dimethoate, malathion, monocrotophos and chlorpyrifos) and carbamate (carbaryl and carbofuran) insecticides. The residues of organochlorine (OC) insecticides did not exceed the Maximum Residue Limit laid down by FAO/WHO in any of the samples. Aldrin up to 0.050 µg g<sup>-1</sup> detected in okra fruits. Similarly, the residues of OP

insecticides were detected in okra fruits but remained below MRL. The residues of  $\beta$ -HCH (OC), permethrin (synthetic pyrethroid), phosphamidon, quinalphos and triazophos (organo phosphate) were not detected in okra fruits.

Tahiret *et al.* (2009) determined the pesticide residues in market samples of fruits and vegetables from Pakistan and revealed that most of the samples were free from pesticide residues of nine selected pesticides (diazinon, carbaryl, chlorpyrifos, imidacloprid, endosulfan, cypermethrin, cyfluthrin, deltamethrin and metalaxyl) but only two samples of tomatoes were found contaminated with imidacloprid, which were within limits set by the WHO.

Anwar *et al.* (2011) determined pesticide residues in eight fruit samples of apple, guava, grapes, orange, pear, persimmon, banana and peach procured from the local markets of Nawabshah, district, Sindh by capillary gas chromatograph equipped with ECD. Dimethoate was recorded in the quantity of 0.042 mg Kg<sup>-1</sup>, 0.090 mg Kg<sup>-1</sup>, 0.139, mg Kg<sup>-1</sup> 0.003 mg Kg<sup>-1</sup>, 0.042 mg Kg<sup>-1</sup> in apple, guava, orange, pear and persimmon respectively. Cypermethrin was found in the quantity of 0.940 mg Kg<sup>-1</sup>, 0.030 mg Kg<sup>-1</sup> and 0.031, mg Kg<sup>-1</sup> in apple, persimmon and peach respectively. Fenvalerate was found in the quantity of 0.055 mg Kg<sup>-1</sup>, 0.002 mgKg<sup>-1</sup>, 0.021 mg Kg<sup>-1</sup>, 0.021 mg Kg<sup>-1</sup> and 0.455 mg Kg<sup>-1</sup> in guava, orange, persimmon and peach respectively. Deltamethrin was recorded in the quantity of 0.039 mgKg<sup>-1</sup>, 0.020 mg Kg<sup>-1</sup> and 0.005 mg in apple, guava and pear respectively.

Jallowet *et al.* (2017) studied the level of pesticide residues in commonly consumed fruits and vegetables in Kuwait. A total of 150 samples of different fresh vegetables and fruits were

analyzed for the presence of 34 pesticides using the quick, easy, cheap, effective, rugged and safe (QuEChERS) multi-residue extraction method, followed by gas chromatography-mass spectrometry (GC-MS) or liquid chromatography-tandem mass spectrometry (LC-MS/MS). Pesticide residues above the maximum residue limits (MRL) were detected in 21 per cent of the samples and 79 per cent of the samples had no residues of the pesticides surveyed or contained residues below the MRL. Multiple residues were present in 40 per cent of the samples with two to four pesticides, and four samples were contaminated with more than four pesticide residues. Of the pesticides investigated, 16 were detected, of which imidacloprid, deltamethrin, cypermethrin, malathion, acetamiprid, monocrotophos, chlorpyrifos-methyl, and diazinon exceeded their MRLs.

Akhtaret *al.* (2018) carried out the study to determine residue of selected pesticides *viz.*, bifenthrin, difenoconazole, paraquat, dimethomorph, imidacloprid, deltamethrin in fruit (guava) and vegetables (eggplant and round gourd) collected from shops in commercial market, Lahore. These samples were prepared and subjected to high pressure liquid Chromatography (HPLC) for detection of pesticide residues. The results showed that in Guava fruit, concentration of bifenthrin, difenoconazole, paraquat, diomethomorph and imidacloprid were 5.13, 81.5, 6.6, 0.48 and 1.65 mg/kg respectively. In egg plant sample, bifenthrin, difenoconazole, paraquat, diomethomorph and deltamethrin detected residues of 3.53, 5.62, 4.58, 0.25 and 0.005 mg/kg respectively while imidacloprid residues was not detected.



### 2.1.2 Lambda cyhalothrin

Hem *et al.* (2010) monitored the residual levels of four insecticidal compounds (lambda-cyhalothrin, lufenuron, thiamethoxam, and clothianidin) in the pomegranate, in order to assess the risk to consumers posed by the presence of such residues. The insecticides were applied at the recommended rates onto pomegranate trees. The samples were then collected at harvesting time after several treatments (two, three, and four treatments). After sample preparation progressed through the clean-up procedure, lufenuron, thiamethoxam, and clothianidin residues were analyzed via a HPCL-UVD, and the lambda-cyhalothrin residue was analyzed via a GC- $\mu$ ECD. The versatility of this method was evidenced by its excellent linearity ( $>0.9998$  to 1) at broad concentration ranges. The mean recoveries evaluated from the untreated sample spiked with two different fortification levels ranged from 72.45 to 113.90 per cent, and the repeatability (as a relative standard deviation) resulted from triplicate recovery tests was in a range from 0.80 to 11.75 per cent. The residues of all insecticides determined from treated pomegranate samples and their LOD levels (lufenuron, 0.01; lambda-cyhalothrin, 0.005; thiamethoxam, 0.01; clothianidin, 0.02 mg/kg) were much lower than their MRLs (0.5 mg/kg).

Gupta *et al.* (2015) studied dissipation and decontamination of imidacloprid and lambda cyhalothrin residue in brinjal. Residues of imidacloprid at 20 g a.i. ha<sup>-1</sup> and lambda cyhalothrin at 15 g a.i. ha<sup>-1</sup> were estimated on brinjal fruits. Initial deposits of imidacloprid dissipated to 93.17 per cent on 10<sup>th</sup> day. In lambda cyhalothrin the initial deposits were 0.138 mg kg<sup>-1</sup> which dissipated to 92.75 per cent on 10<sup>th</sup> day. Both imidacloprid and lambda cyhalothrin had half-life values of 1.92

and 2.65 days, respectively on brinjal fruits. The safe waiting period for imidacloprid was found to be 4.70 days. In lambda cyhalothrin, no waiting period is required after its application as the initial deposits were less than its MRL (0.3 mg kg<sup>-1</sup>).

Djouakaet *al.* (2018) monitored  $\lambda$ -cyhalothrin residues in lettuce and cabbage from farms at the market gates in Cotonou and Parakou using high performance liquid chromatography (HPLC) analysis techniques. These residues were also monitored on samples directly from farms (on-farm sampling) for 14 days post-treatment. Results revealed that the level of  $\lambda$ -cyhalothrin residue concentrations in lettuce from Houeyiho decreased from 4.2 mg/kg on Day 1 to about 0.2 mg/kg on Day 7. On Day 9, analyzed lettuces were all  $\lambda$ -cyhalothrin free. In contrast, even 14 days after treatment of cabbage from Bawera (Parakou), the presence of  $\lambda$ -cyhalothrin residues in analyzed samples were recorded. For samples from market gates,  $\lambda$ -cyhalothrin residues were found in lettuce from two markets out of the nine surveyed in Cotonou. Interestingly, none of these contaminated samples had residues above the maximum residue limit for lettuce (MRL = 0.5 mg/kg). Similarly, in Parakou, samples from all five surveyed vegetable markets were contaminated with  $\lambda$ -cyhalothrin residues at concentrations below the MRL for cabbage (MRL=0.2mg/kg).

Okediranet *al.* (2019) evaluated pesticide residues in fresh vegetables from Mile 12 market in Lagos and verified compliance of these fresh vegetables with the maximum residue levels (MRLs) as specified by Codex Alimentarius Commission. The residues were extracted by means of multi residue method QuEChERS GC-MS was used to determine two organophosphates (dichlorvos and chlorpyrifos), two

organochlorines (endosulfan II and chlorothalonil), two carbamates (carbaryl and carbofuran) and two pyrethroids (alpha-cypermethrin and lambda-cyhalothrin). Fifteen samples of five common vegetables (cabbage, lettuce, fluted pumpkin, bitter leaf and African spinach) were analyzed. Lambda-cyhalothrin was detected in all the five vegetable samples from Mile 12 market in the order of cabbage (0.171 mg/kg)>lettuce (0.118 mg/kg)> fluted pumpkin (0.113 mg/kg)> bitter leaf (0.075 mg/kg)>African spinach (0.064 mg/kg) and were all below the Codex MRL of 0.2mg/kg.

### **2.1.3 Imidacloprid**

Dubaleet *et al.* (2011) carried out field experiment on Alphonso mango fruits to study the residues of cypermethrin, endosulfan, imidacloprid, methyl demeton, quinalphos and monocrotophos against mango hoppers. All the insecticides were applied once on mango trees at recommended dose and suggested that, the residues of above insecticides at 75 and 85 per cent maturity of fruits were below detectable limits. However, the fruits collected at harvesting did not contain any detectable amount of insecticidal residue and hence safe for human consumption.

Banerjee *et al.* (2012) studied persistence of imidacloprid and beta cyfluthrin in vegetables. Insecticides were applied through a ready-mix formulation, Solomon 300 OD at the concentration of 200 and 400 ml ha<sup>-1</sup> on the fruits of brinjal, tomato and okra. The study indicated that the half-life value of beta cyfluthrin was 1.07 and 2.41 days while that of imidacloprid was 1.98 and 3.30 days respectively, suggesting that the persistence of beta cyfluthrin was lower than that of imidacloprid in fruits of these vegetables. Moreover, the

persistence of these insecticides when compared between different fruits, it was highest in brinjal followed by tomato and least in okra.

Kadam *et al.* (2012) studied residues and dissipation of imidacloprid in pomegranate fruits. Imidacloprid was sprayed on pomegranate trees at an interval of 15 days at 27 and 54 g a.i. ha<sup>-1</sup>. The study revealed that residues of imidacloprid persisted upto 3 and 5 days in arils, 5 and 7 days in whole fruits and 7 and 10 days in peel of pomegranate fruits at recommended (27 g a.i. ha<sup>-1</sup>) and higher dose (54 g a.i. ha<sup>-1</sup>), respectively.

Reddy *et al.* (2013) studied residue dynamics of imidacloprid and hexaconazole on mango. Three sprays of imidacloprid 17.8 SL at 125 ml and 250 ml ha<sup>-1</sup> while hexaconazole 5 SL at 100 ml and 200 ml ha<sup>-1</sup> 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. 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 were 4.27 and 5.19 days respectively, after last spray while the waiting periods for hexaconazole on mango at single and double dose were 7.62 and 10 days respectively after last spray.

Jacobo *et al.* (2014) diagnosed pesticide residues in oranges from Nuevo Leon, México, in citrus orchards. Hundred orange fruit samples were collected randomly from orchards and subjected to analysis for 93 pesticides at residual level by GC/MS and LCQ-TOF-MS. Results showed that in 56 per cent

of the samples, pesticide residues were below the LOQ of 0,005 mg/kg. In total, 15 pesticides were found above LOQ; malathion, spiroticlofen, chlorpyrifos-ethyl, carbendazim, imidacloprid, pyraclostrobin, methidathion, dimethoate, thiabendazol, omethoate, thiofanate-methyl, diflubenzuron, methalaxyl, parathion-methyl and pyridaben.

Phartial and Shrivastava (2014) studied dissipation of imidacloprid on lemon fruit. Imidacloprid 17.8 SL was applied at the rate of 0.008 per cent on citrus fruits. Mean initial deposits was found to be 2.66 mg kg<sup>-1</sup> on the rind after first spraying and it was dissipated up to 31.95 per cent in 3 days and 79.32 per cent after 7 days respectively. Residue of Imidacloprid was dissipated up to 91.35 per cent after 11 days of first spray and it was Below Detectable Level (BDL) after 15 days of first spraying. Following second spray, mean initial deposits on the rind was observed to be 3.02 mg kg<sup>-1</sup> and it was dissipated up to 39.07 per cent in 3 days and 80.13 per cent after 7 days of spraying. After 11 days of second spray 89.07 percent residue was dissipated and it reached to below detection level after 15 days of spray.

Gupta et al. (2015) studied dissipation and decontamination of imidacloprid and lambda cyhalothrin residue in brinjal. Residues of imidacloprid at 20 g a.i. ha<sup>-1</sup> and lambda cyhalothrin at 15 g a.i. ha<sup>-1</sup> were estimated on brinjal fruits. Initial deposits of imidacloprid dissipated to 93.17 per cent on 10th day. In lambda cyhalothrin the initial deposits were 0.138 mg kg<sup>-1</sup> which dissipated to 92.75 per cent on 10th day. Both imidacloprid and lambda cyhalothrin had half-life values of 1.92 and 2.65 days, respectively on brinjal fruits. The safe waiting period for imidacloprid was 4.70 days. In lambda cyhalothrin, no

waiting period is required after its application as the initial deposits were less than its MRL (0.3 mg kg<sup>-1</sup>).

Panditet *et al.* (2016) studied persistence and dissipation of imidacloprid in okra, *Abelmoschus esculentus* L. Residue analysis on okra leaf, fruit and in field soil after application of imidacloprid at recommended (T1: 24.5 g a.i. ha<sup>-1</sup>) and double the recommended (T2: 49.0 g a.i. ha<sup>-1</sup>) doses along with control (T3: 0) showed that only 1 day after application (DAA) the residue in okra leaf dissipated to 38-48 per cent and in fruit to 31-44 per cent in T1 and T2 respectively. The residues declined to below detection level (BDL) within 15-20 DAA in leaves and 7 DAA in fruits. The half-life ( $t_{1/2}$ ) ranged between 2.66-3.28 days in leaf and 1.76-2.07 days in fruit. It was revealed that imidacloprid is a safe insecticide to be used in vegetables like okra. The residue level went below MRL (0.5 mg kg<sup>-1</sup> for okra) within 7 days at the recommended dose. In soil, the residue was below the detectable level after 1 and 3 DAA.

Sharma *et al.* (2018) carried out experiment under protected cultivation at CSK Himachal Pradesh KrishiVishvavidyalaya, Palampur. Single spray of three insecticides *viz.*, imidacloprid (0.018%), indoxacarb (0.015%) and lambda-cyhalothrin (0.004%) was given on tomato crop. The residues of imidacloprid and indoxacarb were worked out on tomato fruits during winter season whereas lambda-cyhalothrin residues were worked out during summer season. Tomato fruit samples were collected at 0, 1, 3, 5, 7 and 10 days after application of insecticides. The residues were analyzed using High Performance Liquid Chromatography. The initial deposits of imidacloprid, indoxacarb and lambda-cyhalothrin on tomato fruits were 0.643, 0.401 and 0.550 mg/kg, respectively. The

residues of all the insecticides were below detectable limits on 10<sup>th</sup> day of application. The half-life values of imidacloprid, indoxacarb and lambda-cyhalothrin were 2.91, 5.26 and 3.06 days, respectively. The corresponding safety intervals were worked out to be 0.36, 0.46 and 1.03 days for imidacloprid, indoxacarb and lambda-cyhalothrin.

#### **2.1.4 Thiamethoxam**

Karmarkar and Kulshrestha (2009) carried out field experiment to study persistence of thiomethaxam in tomato fruits. Foliar application of thiamethoxam at the recommended rate of 140 g a.i. ha<sup>-1</sup> and the double rate of 280 g a.i. ha<sup>-1</sup> was done on the crop at fruit initiation stage. Samples were collected at 0 (3 h), 1, 3, 5, 7, 10, 15 and 20 days after spraying. The insecticide dissipated from 82 to 87 per cent in 10 days with a half-life of 4 days. Total residues reached below detectable level in 15 days in tomato fruits.

Hem et al. (2010) monitored the residual levels of four insecticidal compounds (lambda-cyhalothrin, lufenuron, thiamethoxam, and clothianidin) in the pomegranate, in order to assess the risk to consumers posed by the presence of such residues. The insecticides were applied at the recommended dose rates onto pomegranate trees. The samples were then collected at harvesting time after several treatments (two, three, and four treatments). After sample preparation progressed through the clean-up procedure, lufenuron, thiamethoxam, and clothianidin residues were analyzed via HPCLUVD, and the lambda-cyhalothrin residue was analyzed via GC- $\mu$ ECD. The versatility of this method was evidenced by its excellent linearity ( $>0.9998$  to 1) at broad concentration ranges. The mean recoveries evaluated from the untreated sample spiked with two different fortification

levels ranged from 72.45 to 113.90 per cent, and the repeatability (as a relative standard deviation) resulted from triplicate recovery tests in a range from 0.80 to 11.75 per cent. The residues of all insecticides determined from treated pomegranate samples and their LOD levels (lunfenuron, 0.01; lambda-cyhalothrin, 0.005; thiamethoxam, 0.01; clothianidin, 0.02 mg/kg) were much lower than their MRLs (0.5 mg/kg).

Munj (2011) carried out an experiment to determine pre harvest interval of thiamethoxam in Alphonso mango fruits. Thiamethoxam was applied at the spray concentration of 0.0025 per cent on mango trees about two months before harvesting. Mango fruits were harvested for residue study at an interval of 6 days, 12 days, 20 days, 30 days, 35 days, 42 days, 48 days and 60 days after spray application. He reported that mango fruits were free from thiamethoxam residue at 12 days after spray. Thus, he suggested 12 days of pre harvest interval for thiamethoxam.

Xiuguoet *al.* (2013) conducted the experiment in which thiamethoxam 25 WDG when sprayed at two dosages of 0.67 and 1.0 g a.i. ha<sup>-1</sup> in tobacco resulted in 0.020-0.541 mg kg<sup>-1</sup> of harvest time residues in cured tobacco 19 leaves and 0.005-0.019 mg kg<sup>-1</sup> in soil. The average recoveries for green and cured tobacco leaves and soil ranged from 89.7 to 94.8 per cent, 90.6 to 94.4 per cent and 89.0 to 92.8 per cent, respectively. The half-life values ranged from 3.9 to 4.4 days in green tobacco leaves and 12.0 to 19.1 days in soil. The LOD and LOQ were determined as 0.003 and 0.01 mg kg<sup>-1</sup> in tobacco leaf, whereas 0.002 and 0.005 mg kg<sup>-1</sup> in soil, respectively.

Alrahman (2014) carried out field experiments to study residue and dissipation kinetics of thiamethoxam in potato.



Thiamethoxam (Actara 25% WG) was sprayed at the recommended dose (20 g 100 L<sup>-1</sup> water). The average residue of thiamethoxam was 2.16 mg kg<sup>-1</sup> after 1 day of application. The Residues were decreased with the time. The residues were dissipated to an extent of 49 per cent after 3 days showing residues of 1.06 mg kg<sup>-1</sup>. Following that period the residual amount of thiamethoxam dissipated by 78.7 per cent and 100 per cent after 5 and 7 days, respectively. Finally, the residue of thiamethoxam in potato tubers was below its MRL 0.5 mg kg<sup>-1</sup> after 5 days of its application at the recommended dosage.

Bhattacharjee and Dikshit (2016) conducted experiment using thiamethoxam and dimethoate to control hoppers and inflorescence midges in mango. Thiamethoxam (0.008 and 0.016 %) and dimethoate (0.06 and 0.12 %) were sprayed on Dashehari mango trees during the pre-mature stage of fruit (first week of May) to study their dissipation kinetics and risk assessment in mango fruit. Thiamethoxam dissipated in fruit from 1.93 and 3.71 mg kg<sup>-1</sup> after 2 h of spraying to 0.08 and 0.13 mg kg<sup>-1</sup> after 20 days of spraying at single and double doses, respectively. Its residue did not persist beyond 20 days in fruit. Dimethoate dissipated in fruit from 2.81 and 5.34 mg kg<sup>-1</sup> after 2 h of application to 0.12 and 0.19 mg kg<sup>-1</sup> after 10 days of application at single and double doses, respectively. No residue was detected in fruit beyond 10 days after its application. Both ready-to-harvest mature mango fruit and pulp (after 40 days of spraying) were free from any residues of these insecticides at both the concentration levels. The rate of dissipation of these insecticides followed first-order kinetics in fruit with residual half-lives of 4.0 to 4.5 days for thiamethoxam and 2 days for dimethoate. Based on their MRL values of 0.5 and 2.0 mg kg<sup>-1</sup>

in mango, pre-harvest intervals of 7 and 11 days, and 6 and 7 days were suggested for thiamethoxam and dimethoate, respectively, after spraying at single and double doses. The theoretical maximum residue contribution (TMRC) values for both the insecticides, calculated for residues corresponding to each sampling date, were found to be below the maximum permissible intake (MPI) values on mango fruit (except for dimethoate double dose up to 3 days); hence, both thiamethoxam and dimethoate could be considered non-hazardous to consumers at the above doses and time intervals.

Ramadan *et al.* (2016) carried out study to investigate the residual levels and dissipation behavior of four insecticides, *viz.*, abamectin, thiamethoxam, spinosad, and chlorpyrifos, in tomato (*Solanum lycopersicum* L.) fruits under Egyptian field condition. The insecticide residues were determined after application of insecticides for three times at recommended rates. The extraction of insecticide residues was carried out by using QuEChERS method. The determination of residual levels was performed by high performance liquid chromatography coupled with diode array detector (HPLC-DAD). Recoveries were between 85 per cent and 130 per cent, with relative standard deviations from 1.8 per cent to 17.0 per cent at two fortified levels. The initial deposits were 0.255, 4.28, 0.205, and 0.647 mg kg<sup>-1</sup> for abamectin, chlorpyrifos, spinosad, and thiamethoxam, respectively. Residue levels of chlorpyrifos and thiamethoxam decreased to reach 0.326 and 0.03 mg kg<sup>-1</sup> after 15 D from application indicating that 92.4 per cent and 95.4 per cent of chlorpyrifos and thiamethoxam dissipated, respectively.

Rabea *et al.* (2018) studied dinotefuran and thiamethoxam residue levels in pepper samples which were collected randomly

at 0, 1, 3, 5, 7, 10, 15 and 21 days after treatment using QuEChERS method and clean up step utilizing solid phase extraction (SPE) followed by determination by high-performance liquid chromatography with diode-array detection (HPLC/DAD). Dinotefuran and thiamethoxam were recovered within 77-80 per cent and 78-112 per cent, respectively at the spike levels (0.01 - 1 mg/kg) in pepper samples with relative standard deviations (RSDs) lower than 3 per cent. Good linearity was achieved for dinotefuran and thiamethoxam with an excellent correlation coefficient of  $R^2 > 0.996$  and the matrix matched calibration also showed good linearity with determination coefficients  $R^2 > 0.98$ . The initial deposits of dinotefuran and thiamethoxam in pepper fruits were 6.59 and 1.38 mg/kg, respectively. The half-life period (RL50) of tested pesticides on pepper fruits were 2 and 3.11 days for dinotefuran and thiamethoxam, respectively. According to maximum residue level (MRL) (0.01 mg/kg for dinotefuran and 0.7 mg/kg for thiamethoxam) the pre-harvest interval (PHI) was 11 and 4 days, respectively.

### **2.1.5 Dimethoate**

Gupta *et al.* (1998) studied 27 samples of bottle gourd, out of which 4 showed monocrotophos residues in the range of 0.18-0.67 mg/kg, 3 samples were contaminated with fenvalerate, 2 each with endosulfan and phosphamidon and one each with HCH and DDVP. Cauliflower samples were found contaminated with monocrotophos (0.12-1.5 µg/g), quinalphos (0.15-0.6 µg/g), dimethoate (0.05-0.2 µg/g), chlorpyrifos (0.15 µg/g), fenvalerate (0.08-2.5 µg/g), endosulfan (0.05-2.5 µg/g) and HCH (0.03-2.1 µg/g).

Presence of pesticide residues in fruits and vegetables were also reported from Islamabad, Pakistan (Tahiret *et al.* 2001).

Dimethoate was present in the quantity of 0.032 mg/kg in apple, 0.110mg/kg in banana, 0.004 mg/kg in brinjal, 1.80 mg/kg in cauliflower and 0.13 mg/kg in arvi, fenvalerate 0.010 mg/kg in apple and chlorpyrifos 0.004 mg/kg in brinjal.

Arora (2008) analyzed samples of okra and brinjal fruits, collected from non-integrated pest management (Non-IPM) and IPM fields in village Raispur, Ghaziabad District (U.P.), for pesticide residues. During first year of study, the residues of chlorpyrifos and cypermethrin in okra fruit were observed to be 5.75 and 0.625  $\mu\text{g/g}$ , respectively, for non-IPM fields; and 0.104  $\mu\text{g/g}$  of chlorpyrifos for IPM fields. The pesticide residues were found to be 0.77, 1.39, 0.4 and 0.32  $\mu\text{g/g}$  for cypermethrin, chlorpyrifos, monocrotophos and dimethoate, respectively for non-IPM okra fruits in second year. For brinjal fruit, residues of cypermethrin and imidacloprid were not detected in IPM trials while it was found to be 0.28 and 0.78  $\mu\text{g/g}$  for cypermethrin and chlorpyrifos respectively, for non-IPM fields.

Anwar *et al.* (2011) determined pesticide residues in eight fruit samples of apple, guava, grapes, orange, pear, persimmon, banana and peach procured from the local markets of Nawabshah, district, Sindh by capillary gas chromatograph equipped with ECD. Dimethoate was recorded in the quantity of 0.042 mg Kg<sup>-1</sup>, 0.090 mg Kg<sup>-1</sup>, 0.139, mg Kg<sup>-1</sup> 0.003 mg Kg<sup>-1</sup>, 0.042 mg Kg<sup>-1</sup> in apple, guava, orange, pear and persimmon respectively. Cypermethrin was found in the quantity of 0.940 mg Kg<sup>-1</sup>, 0.030 mg Kg<sup>-1</sup> and 0.031, mg Kg<sup>-1</sup> in apple, persimmon and peach respectively. Fenvalerate was found in the quantity of 0.055 mg Kg<sup>-1</sup>, 0.002 mgKg<sup>-1</sup>, 0.021 mg Kg<sup>-1</sup> 0.021 mg Kg<sup>-1</sup> and 0.455 mg Kg<sup>-1</sup> in guava, orange, persimmon and peach respectively. Deltamethrin was recorded in the quantity of 0.039

mgKg<sup>-1</sup>, 0.020 mg Kg<sup>-1</sup> and 0.005 mg in apple, guava and pear respectively.

Jacobo *et al.* (2014) diagnosed pesticide residues in oranges from Nuevo Leon, México, in citrus orchards. Hundred orange fruit samples were collected randomly from orchards and subjected to analysis for 93 pesticides at residual level by GC/ QQQ-MS and LCQ-TOF-MS. Results showed in 56 per cent of the samples, pesticide residues were below the LOQ of 0,005 mg/kg. In total, 15 pesticides were found above LOQ; malathion, spiroticlofen, chlorpyrifos-ethyl, carbendazim, imidacloprid, pyraclostrobin, methidathion, dimethoate, thiabendazol, omethoate, thiofanate-methyl, diflubenzuron, methalaxyl, parathionmethyl and pyridaben.

Bhattacharjee and Dikshit (2016) conducted experiment using thiamethoxam and dimethoate to control hoppers and inflorescence midges in mango. Thiamethoxam (0.008 and 0.016 %) and dimethoate (0.06 and 0.12 %) were sprayed on Dashehari mango trees during the pre-mature stage of fruit (first week of May) to study their dissipation kinetics and risk assessment in mango fruit. Thiamethoxam dissipated in fruit from 1.93 and 3.71 mg kg<sup>-1</sup> after 2 h of spraying to 0.08 and 0.13 mg kg<sup>-1</sup> after 20 days of spraying at single and double doses, respectively. Its residue did not persist beyond 20 days in fruit. Dimethoate dissipated in fruit from 2.81 and 5.34 mg kg<sup>-1</sup> after 2 h of application to 0.12 and 0.19 mg kg<sup>-1</sup> after 10 days of application at single and double doses, respectively. No residue was detected in fruit beyond 10 days after its application. Both ready-to-harvest mature mango fruit and pulp (after 40 days of spraying) were free from any residues of these insecticides at both the concentration levels. The rate of dissipation of these

insecticides followed first-order kinetics in fruit with residual half-lives of 4.0 to 4.5 days for thiamethoxam and 2 days for dimethoate. Based on their MRL values of 0.5 and 2.0 mg kg<sup>-1</sup> in mango, pre-harvest intervals of 7 and 11 days, and 6 and 7 days were suggested for thiamethoxam and dimethoate, respectively, after spraying at single and double doses. The theoretical maximum residue contribution (TMRC) values for both the insecticides, calculated for residues corresponding to each sampling date, were found to be below the maximum permissible intake (MPI) values on mango fruit (except for dimethoate double dose up to 3 days); hence, both thiamethoxam and dimethoate could be considered nonhazardous to consumers at the above doses and time intervals.

## **2.2 To estimate the insecticidal residue from farmers orchards**

Kumari et al. (2002) determined the magnitude of pesticidal contamination in six popularly grown summer vegetables (okra, smooth gourd, bitter gourd, cucumber, tomato and brinjal) in Haryana and found that all the 60 market samples of vegetables were found to be contaminated with 3 or 4 insecticides of each chemical groups i.e. organochlorine, organophosphorus, synthetic pyrethroid and carbamate. Okra fruits were contaminated with organochlorine (HCH, DDT, endosulfan and aldrin), synthetic pyrethroids (cypermethrin, deltamethrin and fenvalerate), organophosphorus (dimethoate, malathion, monocrotophos and chlorpyrifos) and carbamate (carbaryl and carbofuran) insecticides. The residues of organochlorine (OC) insecticides did not exceed the Maximum Residue Limit laid down by FAO/WHO in any of the samples.

Aldrin up to 0.050  $\mu\text{g g}^{-1}$  detected in okra fruits. Similarly, the residues of OP insecticides were detected in okra fruits but remained below MRL. The residues of  $\beta\text{HCH}$  (OC), permethrin (synthetic pyrethroid), phosphamidon, quinalphos and triazophos (organo phosphate) were not detected in okra fruits.

Vijayalakshami (2002) conducted the experiment in which the initial deposition of quinalphos @ 0.05 per cent in/on mango fruits was 3.76 ppm which dissipated to 0.14 ppm with 96 per cent dissipation after 15 days. Furthermore, at 0.2 per cent concentration residue was 6.75 ppm which reduced to 0.23 ppm with 97 per cent dissipation after 15 days. Therefore, she reported waiting periods of 11.5 and 14.2 days after spray for the safe consumption of fruits at 0.05 and 0.1 per cent dose, respectively before harvest.

Hassan *et al.* (2005) carried out an experiment to study persistence of chlorpyrifos, imidacloprid and acephate in brinjal fruits. The residues of chlorpyrifos in brinjal fruits, 3 h after spraying, were found to be 0.075 ppm which dissipated to 0.050 and 0.039 ppm at 3 and 7 days respectively. Imidacloprid residues in brinjal fruit, 3 h after spraying were 0.038 ppm which degraded to 0.020 and 0.015 ppm at 3 and 7 days respectively. The residues of acephate at 3 h after spraying were 0.067 ppm which dissipated to 0.051 and 0.04 ppm in 3 and 7 days respectively. This leads to conclusion that, it is safe to consume brinjal fruits after three days of spraying of above insecticides.

Arora *et al.* (2006) from Uttar Pradesh, monitored the mango samples for chlorpyrifos and monocrotophos from IPM and non-IPM plots wherein, the residues of chlorpyrifos in pulp, peel and whole mango fruit were 0.218, 0.198 and 0.452  $\mu\text{g g}^{-1}$

in IPM as well as 0.609, 0.589 and 1.326  $\mu\text{g g}^{-1}$  in non-IPM plots, respectively. Furthermore, the residues of monocrotophos were not detected in both IPM and non-IPM plots of mango.

Mukherjee *et al.* (2007) from Uttar Pradesh, revealed that marketable mango samples collected from farmer's IPM plots did not show any trace of insecticide residues except  $\alpha$ -endosulfan (0.04 mg/kg) and  $\beta$ -endosulfan (0.05 mg/kg) when analyzed for chlorpyrifos, methyl parathion,  $\alpha$ -endosulfan,  $\beta$ -endosulfan, endosulfansulphate, cypermethrin and fenvalerate.

Arora *et al.* (2008) conducted a field experiment to study dissipation of imidacloprid on kinnow mandarin fruits. Two applications of imidacloprid were done at weekly interval both for recommended and double recommended dose i.e. 0.008 and 0.016 per cent. The average initial deposits at both these concentrations on the rind of kinnow mandarin fruits were found to be 2.40 and 3.90 mg  $\text{kg}^{-1}$  respectively. The corresponding values in the pulp were observed to be 0.03 and 0.04 mg  $\text{kg}^{-1}$ , respectively. A sudden decline in the residue levels in the rind and a corresponding increase in the pulp was observed on 5<sup>th</sup> day, thereafter, the residue levels steadily decreased both in the rind and pulp. Residues of imidacloprid were below its maximum residue limit in whole fruits at zero day sampling.

From Pakistan, Tahiret *et al.* (2009) determined the pesticide residues in market samples of fruits and vegetables and revealed that most of the samples were free from pesticide residues of nine selected pesticides (diazinon, carbaryl, chlorpyrifos, imidacloprid, endosulfan, cypermethrin, cyfluthrin, deltamethrin and metalaxyl) but only two samples of tomatoes were found contaminated with imidacloprid, which were within limits set by the WHO.



Mahdavian and Somashekhar (2010) procured twenty two samples of grapes from Bangalore city market and analyzed for synthetic pyrethroids like cypermethrin and fenvalerate. The results showed that all the samples were contaminated with synthetic pyrethroids beyond the MRL as defined by the FAO/WHO and grapes committee.

Mohapatra and Ahuja (2010) evaluated persistence of lambda cyhalothrin residue in mango fruits. Repeated spray applications of lambda cyhalothrin (Karate 5 EC) at the recommended dose of 0.5 and double recommended dose 1.0 ml/litre, resulted in the initial deposit of 0.86 and 1.6 mg/kg, respectively. Moreover, the residues dissipated steadily and after 20 days of spraying of 0.05 and 0.09 mg/kg were recovered from the fruit peel and fruit pulp and maximum of 0.034 and 0.044 mg/kg respectively. Lambda cyhalothrin was recovered after 5 days, following treatment at recommended and double dose, respectively. They further reported that lambda cyhalothrin residues in mango peel dissipated at the half-life of 4.8 days and the safe pre-harvest interval was 4 and 8 days taking the maximum residue limit value as 0.5 mg/kg.

Pujeriet *al.* (2010) from Karnataka, monitored the pesticide residues in pomegranate and showed that out of five samples, four samples were found contaminated with pesticides like cypermethrin, dichlorovos, malathion, monocrotophos, and hexaconazole. Moreover, the concentration level of these pesticides in pomegranate found above the European Maximum Residue Level values.

Dubaleet *al.* (2010) studied dissipation pattern of cypermethrin in Alphonso mango fruits. Cypermethrin was sprayed once at 0.0075 per cent concentration when the fruits

were at egg size stage. The initial residues of 0.247 ppm dissipated to below MRL of 0.2 ppm within 2.34 days and reached to BDL (0.05 ppm) within 14.92 days. The residual half-life value was 8.28 days. Hence, a waiting period of 2-3 days is required for safe consumption of fruits.

Charan *et al.* (2010) screened the farm gate vegetables samples for pesticide residues and stated that one sample of okra was contaminated with methyl parathion ( $0.22 \mu\text{g g}^{-1}$ ) which exceeded the MRL ( $0.2 \mu\text{g g}^{-1}$ ) among 25 samples. In case of brinjal, four, seven and two samples out of 46 were contaminated with monocrotophos, methyl parathion and cypermethrin respectively.

The monitoring program for different pesticide residues in fruits was carried out at Karachi, Pakistan by Parveen *et al.* (2011). The results showed that one mango sample was found contaminated with lambda-cyhalothrin, whereas same mango sample was free from quinalphos residue, among the various group of pesticides screened for multi-location mango samples.

Anwar *et al.* (2011) determined pesticide residues in eight fruit samples of apple, guava, grapes, orange, pear, persimmon, banana and peach procured from the local markets of Nawabshah, district, Sindh by capillary gas chromatograph equipped with ECD. Dimethoate was recorded in the quantity of  $0.042 \text{ mg Kg}^{-1}$ ,  $0.090 \text{ mg Kg}^{-1}$ ,  $0.139 \text{ mg Kg}^{-1}$ ,  $0.003 \text{ mg Kg}^{-1}$ ,  $0.042 \text{ mg Kg}^{-1}$  in apple, guava, orange, pear and persimmon respectively. Cypermethrin was found in the quantity of  $0.940 \text{ mg Kg}^{-1}$ ,  $0.030 \text{ mg Kg}^{-1}$  and  $0.031 \text{ mg Kg}^{-1}$  in apple, persimmon and peach respectively. Fenvalerate was found in the quantity of  $0.055 \text{ mg Kg}^{-1}$ ,  $0.002 \text{ mg Kg}^{-1}$ ,  $0.021 \text{ mg Kg}^{-1}$ ,  $0.021 \text{ mg Kg}^{-1}$  and  $0.455 \text{ mg Kg}^{-1}$  in guava, orange, persimmon and peach

respectively. Deltamethrin was recorded in the quantity of 0.039 mg Kg<sup>-1</sup>, 0.020 mg Kg<sup>-1</sup> and 0.005 mg in apple, guava and pear respectively.

Mahopatra *et al.* (2012) studied residue dynamics of spirotetramat and imidacloprid in mango fruits. The insecticides were applied as combination formulation, spirotetramat 12 % + imidacloprid 12 % (240 SC) at 90 and 180 g a.i. ha<sup>-1</sup>. Initial residues of imidacloprid on mango fruits from the two treatments were 0.329 and 0.536 mg kg<sup>-1</sup>, respectively. Imidacloprid residues remained on mango fruits beyond 15 days and dissipated with the half-life of 5.2 and 8.2 days.

Shinde *et al.* (2012) studied residues of cypermethrin in okra leaves and fruits. Cypermethrin were applied separately in three different concentrations i.e. 50 ppm, 75 ppm, 100 ppm on okra crops. The residues were determined on 0, 1, 3, 5, 7, 11, 13, 15, 17, 19 and 21 days after application. The initial deposits of 3.619 ppm on leaves and 3.471 ppm on fruits reduced to below detectable level on 17<sup>th</sup> day at concentration of 50 ppm. The residues of 4.019 ppm on leaves and 3.916 ppm on fruits reached to below detectable level after 17 and 19 days at concentration of 75 ppm. The initial residues of 4.531 ppm and 4.219 ppm reached to below detectable level on 21<sup>th</sup> day at concentration of 100 ppm.

Chauhan *et al.* (2013) carried out a field experiment to study dissipation pattern of thiamethoxam on okra fruits. Thiamethoxam was applied at recommended dose i.e. 25 g a.i. ha<sup>-1</sup> at fruiting stage of okra showed that the initial deposits of 0.245 mg kg<sup>-1</sup> reached below detectable level of 0.005 mg kg<sup>-1</sup> at 15 days after application with a half-life period of 1.47 days.

Mahopatra (2014) studied residue dynamics of chlorpyrifos and cypermethrin in/on pomegranate. The residues after treatment on fruit peel were 2.46 and 3.51 mg kg<sup>-1</sup> and 2.84 and 4.54 mg kg<sup>-1</sup> for chlorpyrifos and cypermethrin respectively from recommended and double recommended dose treatment. Chlorpyrifos residues degraded faster as compared to cypermethrin. The pre harvest intervals of chlorpyrifos were 22 and 35 days and those of cypermethrin 50 and 73 days respectively at recommended and double recommended dose treatment.

Chandra *et al.* (2014) studied persistence pattern of chlorpyrifos, cypermethrin and monocrotophos in okra. Insecticides were applied at the dose of 100, 200 and 300 g a.i. ha<sup>-1</sup>. The average initial residues of chlorpyrifos, cypermethrin and monocrotophos were in the range of 0.389-0.874, 0.378-0.862 and 0.391-0.898 mg kg<sup>-1</sup> respectively. The residues of pesticides reached below detection in the 15, 17 and 19 days for chlorpyrifos, cypermethrin and monocrotophos respectively.

Singh *et al.* (2015) studied persistence pattern and risk assessment of cypermethrin residues on chilli following three applications of cypermethrin 25 EC at 50 and 100 g a.i. ha<sup>-1</sup> at 10 days interval. The average initial deposits of cypermethrin in chilli fruits were found to be 1.46 and 3.11 mg kg<sup>-1</sup> at recommended and double recommended dose respectively. Half-life periods of cypermethrin were found to be 4.43 and 4.70 days at single and double dose respectively. Residues declined below its limit of quantification of 0.05 mg kg<sup>-1</sup> after 25 days at both the application doses.

Bhattacharjee and Dikshit (2016) conducted experiment using thiamethoxam and dimethoate to control hoppers and

inflorescence midges in mango. Thiamethoxam (0.008 and 0.016%) and dimethoate (0.06 and 0.12 %) were sprayed on Dashehari mango trees during the pre-mature stage of fruit (first week of May) to study their dissipation kinetics and risk assessment in mango fruit. Thiamethoxam dissipated in fruit from 1.93 and 3.71 mg kg<sup>-1</sup> after 2 h of spraying to 0.08 and 0.13 mg kg<sup>-1</sup> after 20 days of spraying at single and double doses, respectively. Its residue did not persist beyond 20 days in fruit. Dimethoate dissipated in fruit from 2.81 and 5.34 mg kg<sup>-1</sup> after 2 h of application to 0.12 and 0.19 mg kg<sup>-1</sup> after 10 days of application at single and double doses, respectively. No residue was detected in fruit beyond 10 days after its application. Both ready-to-harvest mature mango fruit and pulp (after 40 days of spraying) were free from any residues of these insecticides at both the concentration levels. The rate of dissipation of these insecticides followed first-order kinetics in fruit with residual half-lives of 4.0 to 4.5 days for thiamethoxam and 2 days for dimethoate. Based on their MRL values of 0.5 and 2.0 mg kg<sup>-1</sup> in mango, pre-harvest intervals of 7 and 11 days, and 6 and 7 days were suggested for thiamethoxam and dimethoate, respectively, after spraying at single and double doses. The theoretical maximum residue contribution (TMRC) values for both the insecticides, calculated for residues corresponding to each sampling date, were found to be below the maximum permissible intake (MPI) values on mango fruit (except for dimethoate double dose up to 3 days); hence, both thiamethoxam and dimethoate could be considered nonhazardous to consumers at the above doses and time intervals.

Hafez and Singh (2016) studied persistence of thiamethoxam in/on tomato fruits and soil. Dissipation behavior of thiamethoxam in tomato fruits and soil was studied following

application with recommended dose (50 g a.i. ha<sup>-1</sup>) and double recommended dose (100 g a.i. ha<sup>-1</sup>). The initial deposits in tomato fruits (1 h after spraying) were found to be 0.11 µg g<sup>-1</sup> and 0.18 µg g<sup>-1</sup> at recommended and double recommended dose, respectively. While the initial deposits in soil were found to be 0.06 µg g<sup>-1</sup> and 0.11 µg g<sup>-1</sup> at recommended dose and double recommended dose, respectively. Residues of thiamethoxam reached below determination limit at 7<sup>th</sup> day and 10<sup>th</sup> day in tomato fruits and 7<sup>th</sup> day in soil for recommended and double recommended dose, respectively. The half-life values of tomato fruits were calculated to be 2.21 and 2.41 days for recommended and double recommended doses, respectively.

Sharma *et al.* (2018) carried out experiment under protected cultivation at CSK Himachal Pradesh KrishiVishvavidyalaya, Palampur. Single spray of three insecticides *viz.*, imidacloprid (0.018%), indoxacarb (0.015%) and lambda-cyhalothrin (0.004%) was given on tomato crop. The residues of imidacloprid and indoxacarb were worked out on tomato fruits during winter season whereas lambda-cyhalothrin residues were worked out during summer season. Tomato fruit samples were collected at 0, 1, 3, 5, 7 and 10 days after application of insecticides. The residues were analyzed using High Performance Liquid Chromatography. The initial deposits of imidacloprid, indoxacarb and lambda-cyhalothrin on tomato fruits were 0.643, 0.401 and 0.550 mg/kg, respectively. The residues of all the insecticides were below detectable limits on 10<sup>th</sup> day of application. The half-life values of imidacloprid, indoxacarb and lambda-cyhalothrin were 2.91, 5.26 and 3.06 days, respectively. The corresponding safety intervals were worked out to be 0.36, 0.46 and 1.03 days for imidacloprid, indoxacarb and lambda-cyhalothrin.

### CHAPTER III

## MATERIAL AND METHODS

The present investigation, entitled, “Estimation of insecticidal residue in Alphonso mango” was conducted at Mango Farm, Regional Fruit Research Station, Vengurla and Mango orchards of private farmers in the year 2020. The analytical work was done at Research laboratory of College of Forestry, Dapoli, Dist. Ratnagiri.

### 3.1. To estimate the residue of recommended insecticides in Alphonso mango

#### Experiment Details:

**Experimental site:** Mango Farm, Regional Fruit Research Station, Vengurla Dist. Sindhudurg (M.S.)

**Crop** : Mango

**Variety** : Alphonso

**Design** : R.B.D

**No. of treatments:** 11

**No. of replications** : 3

**Treatment details** :

Tr. No.	Insecticides	Dose ml org/10 lit
1	Deltamethrin 2.8%EC	9
2	Deltamethrin 2.8%EC	18
3	Lambda cyhalothrin 5% EC	6
4	Lambda cyhalothrin 5% EC	12
5	Imidacloprid 17.8 % SL	3
6	Imidacloprid 17.8 % SL	6
7	Thiamethoxam 25 % WG	1
8	Thiamethoxam 25 WG	2
9	Dimethoate 30 % EC	10
10	Dimethoate 30 % EC	20
11	Control (Water Spray)	-

To estimate the residue of above insecticides, 15 year old one mango tree per treatment were selected. Treatment wise insecticide were sprayed on each Mango tree with the help of Gatoor sprayer at 75% fruit maturity (12 ana stage). After spraying, the mangoes were harvested at 1, 5, 10, 15 and 25<sup>th</sup> day as a sample for residual analysis. The extraction, clean up and analysis work of the samples were performed by following standard procedure using HPLC (High Performance Liquid Chromatography) at College of Forestry, Dapoli.

### **3.1.1 Chemicals, glassware's and instruments used for the analysis of pesticide residues**

#### **3.1.1.1 Standards, Solvents and reagents**

Pesticide standards of high purity ( $\geq 98\%$ ) (Technical Grade) supplied by Sigma Aldrich India Ltd., Mumbai were used for the analytical purpose. The obtained vials of pesticide standards were kept according to temperature requirements and used when required for preparation of stock solutions or individual standards. Moreover, different organic solvents and reagents like acetone, n-hexane, acetonitrile, dichloromethane, acetic acid, methanol, chloroform, sodium chloride (saturated), anhydrous sodium sulphate, florisil of HPLC grade and high purity were used for the preparation of standards and extraction and clean up methodologies during the present investigations.

#### **3.1.1.2 Glasswares**

During analysis of pesticide residue the different glasswares like beaker, measuring cylinder, conical flask, volumetric flask, Buchner funnel, test tube (graduated), separatory funnel, glass column and glass rod were used (make



Merck and Borosil). The above mentioned glasswares were washed with acetone and dried at 120°C in oven to avoid cross contamination of pesticides.

### **3.1.1.3 Equipments and instruments**

The details of equipments and instruments used during extraction and clean up methodologies as well as quantitation of pesticide residues during the study period is narrated here under.

**Glassware dryer:** Glassware dryer was used for drying the washed glasswares.

**Homogenizer:** Homogenizer was used for homogenization and fine grinding of sample.

**Analytical balance:** With a view to weigh the samples and reagents accurately, digital weighing balance having lowest sensitivity ( $\pm 0.1$  mg) was used.

**Blender:** Blender was used for the blending the homogenized samples.

**Vortex:** Vortex mixture was used for proper mixing of sample and standards.

**Centrifuge:** Thermo Scientific centrifuge machine was used for the centrifugation of the samples.

**Rotary Vacuum Evaporator:** Rotary Vacuum Evaporator was used to concentrate the large volume of the sample extract.

**HPLC:** Thermo Scientific make High Performance Liquid Chromatograph (HPLC) equipped with PDA (Photo Diode Array) was used.

### 3.1.2 Preparation of pesticide standard solutions

The standards (Technical Grade) of five pesticides *viz.*, deltamethrin, lambda-cyhalothrin, imidacloprid, thiamethoxam and dimethoate with more than 99 per cent purity were used for preparation of standard solutions of different (desirable) concentration for quantitative estimation. In order to run the pesticide standards, individual solutions of both the pesticides were prepared and run on High Performance Liquid Chromatography by using PDA (Photo Diode Array) detector at their variable instrument parameters.

#### 3.1.2.1 HPLC- amenable stock solution

**Deltamethrin:** Stock solution was prepared by weighing 10 mg in 100 ml capacity volumetric flask and dissolved with 100 ml of HPLC grade acetonitrile.

**Lambda-cyhalothrin:** Stock solution was prepared by weighing 10 mg in 100 ml capacity volumetric flask and dissolved with 100 ml of HPLC grade methanol.

**Imidacloprid:** Stock solution was prepared by weighing 20 mg in 100 ml capacity volumetric flask and dissolved with 100 ml of HPLC grade water and acetonitrile in the ratio of 65:35 ml v/v.

**Thiamethoxam:** Stock solution was prepared by weighing 10 mg in 50 ml capacity volumetric flask and dissolved with 25 ml of HPLC grade acetonitrile.

**Dimethoate:** Stock solution was prepared by weighing 25 mg in 50 ml capacity volumetric flask and dissolved with 25 ml of HPLC grade acetonitrile.

$$\text{ppm} = \frac{\text{Weight (g)} \times 10^{-6} \times \text{Purity (of Standard)}}{\text{Volume to be prepared}} \times 100$$

### **3.1.3 Extraction and clean-up of deltamethrin**

A mango sample of 25 grams were crushed or cut 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 chloroform and the washing 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-evaporator was rinsed with two portions of 10 ml of hexane which were also transferred in the column and let to pass; the eluate 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. It is then ready to be analysed.

#### **3.1.3.1 HPLC parameters for deltamethrin**

1. Instrument : High Performance Liquid Chromatography
2. Detector : Photo Diode Array (PDA)
3. Column : C18 dionex
4. Mobile Phase : Water : acetonitrile, HPLC grade (08:92, v/v)
5. Flow rate : 0.85 ml/min
6. Injection volume : 20 µl
7. Wavelength : 233 nm
8. Retention time : 8.0 min

#### **3.1.4 Extraction of lambda-cyhalothrin**

For each sample to be analyzed, about 100 g of mango sample was chopped and mixed. From this mixture, 1 g was weighed and ground with 1 mL HPLC technical grade acetonitrile. The slurry was vigorously shaken on a vortex for 20 min and centrifuged for 20 min at 10,000 rpm for phase separation. The supernatant was collected and re-centrifuged for 15 min at 10,000 rpm; an aliquot of 75 µL was then loaded into HPLC vials for quantification of lambda-cyhalothrin residues.

##### **3.1.4.1 HPLC parameters for lambda-cyhalothrin**

1. Instrument : High Performance Liquid Chromatography
2. Detector : Photo Diode Array (PDA)
3. Column : C18 dionex
4. Mobile Phase : Water : Methanol, HPLC grade (10:90, v/v)
5. Flow rate : 1.0 ml/min
6. Injection volume : 50 µl
7. Wavelength : 226 nm
8. Retention time : 7.20 min

### **3.1.5 Extraction and clean-up of imidacloprid**

The extraction and clean up procedure were followed as suggested by Mohapatra *et. al.* (2012) with certain modifications. The mango fruit samples were macerated in grinder. A representative sample of 50 gm was homogenized with 100 ml acetonitrile in Warring blender and filtered under vacuum through a Buchner funnel. The container and the filter cakes were washed with acetonitrile (2 X 50 ml). The combined extracts were collected in a 500 ml conical flask. The acetonitrile fraction was concentrated (up to 7 ml) under reduced pressure in a rotary vacuum evaporator by keeping 45°C temperature and at 85 rpm. Thus, the obtained aqueous extract was transferred in to 1L separatory funnel along with rinsing of acetonitrile and diluted with 80 ml of distilled water after adding 25 ml saturated sodium chloride solution. The aqueous phase was partitioned with dichloromethane (2 X 50 ml). The combined solvent fraction (100 ml) was dried over anhydrous sodium sulphate, concentrated to 5 ml by using rotary vacuum evaporator and subjected to column chromatography.

#### **Clean up:**

A glass column (1 cm diameter x 30 cm length) packed with 5 g florosil in between 1inch layer of sodium sulphate was used for clean-up. The column was pre-washed with 10 ml HPLC grade acetonitrile. When the solvent front reached the top to the column, the sample extract (5 ml) was transferred by rinsing the test tube in to column. The imidacloprid residues from column were eluted by 50 ml acetonitrile. Thus, collected acetonitrile fraction in flask was concentrated near to dryness by using vacuum rotary evaporator at 45°C temperature and 85 rpm.

Thereafter, the final volume was made with 5 ml HPLC grade acetonitrile and subjected to HPLC analysis.

#### **3.1.5.1 HPLC parameters for imidacloprid**

1. Instrument : High Performance Liquid Chromatography
2. Detector : Photo Diode Array (PDA)
3. Column : C18 dionex
4. Mobile Phase : Water : acetonitrile, HPLC grade (65:35, v/v)
5. Flow rate : 0.7 ml/min
6. Injection volume : 20  $\mu$ l
7. Wavelength : 270 nm
8. Retention time : 4.20 min

#### **3.1.6 Extraction and clean-up of thiamethoxam and dimethoate**

Mango fruit samples (20 g, in triplicate) were homogenized and extracted twice with 50 + 50 mL acetonitrile in a vertical homogenizer after soaking overnight and filtered through a Buchner funnel. Pooled acetonitrile extract was evaporated to near dryness under vacuum in a rotary vacuum evaporator at 50 °C. The remainder was transferred into a 250-mL separating funnel and extracted thrice with hexane (3 × 30 mL) after diluting with 15 per cent sodium chloride solution (50 mL). The hexane layer was discarded and the aqueous layer was partitioned three times with dichloromethane (3 × 30 mL). The pooled dichloromethane extract was dried by passing through anhydrous sodium sulphate and evaporated completely in a rotary vacuum evaporator (40 °C). The residue was then reconstituted with 2 mL HPLC-grade acetonitrile. The same extraction and clean-up method was employed for both thiamethoxam and dimethoate.

### **3.1.6.1 HPLC parameters for thiamethoxam**

1. Instrument : High Performance Liquid Chromatography
2. Detector : Photo Diode Array (PDA)
3. Column : C18 dionex
4. Mobile Phase : Water : acetonitrile, HPLC grade (65:35, v/v)
5. Flow rate : 0.8 ml/min
6. Injection volume : 20 µl
7. Wavelength : 242 nm
8. Retention time : 5.653 ± 0.25 min

### **3.1.6.2 HPLC parameters for dimethoate**

1. Instrument : High Performance Liquid Chromatography
2. Detector : Photo Diode Array (PDA)
3. Column : C18 dionex
4. Mobile Phase : Water : acetonitrile, HPLC grade (60:40, v/v)
5. Flow rate : 1.0 ml/min
6. Injection volume : 20 µl
7. Wavelength : 221 nm
8. Retention time : 4.949 ± 0.03 min

## **3.2. To estimate the insecticidal residue from farmers orchards**

### **Experiment Details:**

**Experimental site:** Mango Farmers from Sindhudurg district

**Crop:** Mango

**Variety:** Alphonso

**Design:** R.B.D.

**No. of treatments:** 4

**No. of replications:** 5

**Treatment details:**

<b>Tr. No.</b>	<b>Treatment</b>
1	Farmers practice up to 5 sprays
2	Farmers practice up to 6 to 10 sprays
3	Farmers practice above 10 sprays
4	Farmers practice with recommended sprays

The farmers from Sindhudurg district as per the treatment were selected. The mangoes of 85 per cent maturity were collected from each farmer. Five samples from east, west, north, south corners and top of the tree of the orchard were taken from each farmer considering one sample as one replication. The samples collected were analyzed for insecticides *viz.*, deltamethrin, lambda cyhalothrin, imidacloprid, thiamethoxam and dimethoate. The extraction, clean up and analysis work of the samples were performed by following standard procedure given above using HPLC (High Performance Liquid Chromatography) at College of Forestry, Dapoli.



## **CHAPTER IV**

### **RESULTS AND DISCUSSION**

The present investigation was carried out in the Konkan region of Maharashtra to study the “Estimation of insecticidal residue in Alphonso mango” in the year 2020. The results obtained during the present investigation are presented with respect to the objectives under the following headings.

- 4.1 To estimate the residue of recommended insecticides in Alphonso mango
- 4.2 To estimate the insecticidal residue from farmers orchards

#### **4.1 To estimate the residue of recommended insecticides in Alphonso mango**

The insecticides were sprayed at recommended and double recommended doses on Mango trees. Mangoes from such treated trees were harvested at 1, 5, 10, 15 and 25<sup>th</sup> day after spraying. The collected mangoes were cut into pieces, blended, prepared extract of it, undergo clean up procedure and such clear sample was injected in High Performance Liquid Chromatography (HPLC) for residue estimation.

Mango samples were collected one day after spray of insecticides and analyzed for the recovery of insecticides in HPLC column. Data of recovery of different insecticides under study in Mango fruits at different levels of concentration one day after spraying are presented in Table1.

**Table 1: Percent mean recovery of pesticides in mango fruits at different levels of concentration on first day after spraying**

Tr. No.	Insecticides	Dose ml org/10 lit	Concentration (ppm)	Limit of detection (LOD)	Limit of Quantification (LOQ)
1	Deltamethrin 2.8%EC	9	0.14	0.01	0.06
2	Deltamethrin 2.8%EC	18	0.20		
3	Lambda cyhalothrin 5% EC	6	0.84	0.03	0.08
4	Lambda cyhalothrin 5% EC	12	1.40		
5	Imidacloprid 17.8% SL	3	0.12	0.02	0.06
6	Imidacloprid 17.8% SL	6	0.16		
7	Thiamethoxam 25% WG	1	1.21	0.01	0.05
8	Thiamethoxam 25 WG	2	2.02		
9	Dimethoate 30 % EC	10	1.92	0.02	0.05
10	Dimethoate 30 % EC	20	3.58		
11	Control (Water Spray)	-	-		

#### 4.1.1 Monitoring Deltamethrin residues in mango fruits

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<sup>th</sup> day and thereafter was not detectable at 10, 15 and on 25<sup>th</sup> day( Table 2). 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 2: 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 deltamethrin sprayed at double than recommended dose (18ml/10 litre) revealed that after one day, double recommended dose of deltamethrin (18 ml/10 lit.) recorded residue of 0.20 ppm which was reduced to 0.12 ppm on 5<sup>th</sup> day and thereafter was not detectable at 10, 15 and on 25<sup>th</sup> 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) 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 3.

**Table 3: 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<sup>th</sup> 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<sup>th</sup> day and it was totally lost on 10<sup>th</sup> 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) and brinjal (SenandChowdhury,1999).

#### **4.1.2 Monitoring Lambda cyhalothrin residues in Mango fruits**

Data on residue of Lambda cyhalothrin sprayed at recommended and double doses 1,5,10,15 and 25 days after spraying are presented in Table 4. The results revealed that after one day, recommended dose of Lambda cyhalothrin (6 ml/10 lit.) showed residue of 0.84 ppm which was reduced to 0.48, 0.21 and 0.04 ppm on 5, 10 and 15 days after spray. On 25<sup>th</sup> day the residue of Lambda cyhalothrin was below detectable level. This indicated that the residue of Lambda cyhalothrin at recommended dose of 6 ml/10 litre lasts up to 15 days and thereafter it is not detectable.

**Table 4: Residue of Lambda cyhalothrin in mango fruits at periodic interval**

<b>Dose ml/10 lit</b>	<b>Average residue (ppm) at periodic intervals (days)</b>				
	<b>1</b>	<b>5</b>	<b>10</b>	<b>15</b>	<b>25</b>
6	0.84	0.48	0.21	0.04	N. D.
12	1.40	0.95	0.53	0.10	N. D.
SE $\pm$	0.03	0.02	0.01	0.02	-
CD at 1 %	0.06	0.04	0.02	0.04	-
Significance	Sig.	Sig.	Sig.	Sig.	

ND: Not Detectable

The results of Lambda cyhalothrin sprayed at double than recommended dose (12ml/10 lit.) revealed that after one day, double recommended dose of Lambda cyhalothrin (12 ml/10 lit.) recorded residue of 1.40 ppm which was reduced to 0.95, 0.53 and 0.10 ppm on 5,10 and 15 days after spraying. The residue of Lambda cyhalothrin at 25 days after spraying was below detectable level. This indicated that the residue of Lambda cyhalothrin at double recommended dose of 12 ml/10 litre lasts up to 15 days and thereafter it is not detectable.

Data on residue of Lambda cyhalothrin at 1,5,10 and 15 days after spraying revealed that the recommended dose 6 ml/10 litre of water was found to be significantly superior over double dose of Lambda cyhalothrin i.e. 12 ml/10 litre.

The results reported by Sharma *et al.*(2018) revealed that the residue of lambda cyhalothrin was below detectable limit (0.03mg/kg) on 10<sup>th</sup> day of spray on tomatosupports present findings.

Dissipation pattern of lambda cyhalothrin in mango fruits was also studied in the present investigation. Data pertaining to dissipation study are presented in Table 5.

Per cent loss of lambda cyhalothrin residue at a dose of 6 ml/10 litre on 5<sup>th</sup> day was 42.83per cent which was reduced to 75.00 and 95.23 per cent at 10 and 15 days after spraying. The residue of lambda cyhalothrin was totally lost at 25 days after spraying. The half-life of lambda cyhalothrin at a dose of 6 ml per 10 litre was observed to be 8.12 days and waiting period was 18 days. The insecticide lambda cyhalothrin at a dose of 12 ml per 10 litre dissipated 32.14 per cent on 5<sup>th</sup> day then it was reduced to 62.14 and 92.85 per cent at 10 and 15 days after

spraying and later on it was totally lost on 25<sup>th</sup> day. The half-life of lambda cyhalothrin at a dose of 12 ml per 10 litre was recorded to be 8.75 days and waiting period was 19 days. This indicated that the higher dose of lambda cyhalothrin dissipates faster than recommended dose. The results of the present investigation revealed that the insecticide lambda cyhalothrin persist for a longer time, this will be useful as far as management of insect pests for a longer time is concerned but it leaves residue for the longer time. The results stating Lambda cyhalothrin residues in mangopeel dissipated at the half-life of 4.8 days reported by Mohapatra and Ahuja (2010) are in conformity with the present findings.

**Table 5: Dissipation pattern of Lambda cyhalothrin from 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		
6	0.00	42.83	75.00	95.23	100	8.12	18
12	0.00	32.14	62.14	92.85	100	8.75	19
SE ±	-	3.76	4.84	6.32	-	-	-
CD at 1 %	-	8.50	N S	N S	-	-	-
Significance	-	Sig.	Sig.				

N S: Not Significant

#### **4.1.3 Monitoring Imidacloprid residues in Mango fruits**

Imidacloprid residue of the dose 3 ml per 10 litre in HPLC on day one after spraying was 0.12 ppm which reduced to 0.06 ppm at 5 days after spraying. It was below detectable level on 10<sup>th</sup> day after spraying. The residue of imidacloprid at double than recommended dose of 6 ml per 10 litre at one day after spraying was

0.16 ppm which was reduced to the extent of 0.10 and 0.04 ppm at 5 and 10 days after spraying and later on it was not detectable (Table 6).

**Table 6: Residue of Imidaclopridin Mango fruits at periodic interval**

Dose ml/10 lit	Average residue (ppm) at periodic interval (days)				
	1	5	10	15	25
3	0.12	0.06	ND	ND	ND
6	0.16	0.10	0.04	ND	ND
SE $\pm$	0.02	0.01	-	-	-
CD at 1 %	-	0.03	-	-	-
Significance	NS	Sig.			

ND: Not Detectable

The results of the present findings are in accordance with the results of Reddy *et al.*(2013) wherein they reported initial deposits of imidacloprid at single and double dose treated mango samples were 0.52 and 0.83 ppm respectively, which dissipated to Below Detectable Level (BDL) at 5 and 7 days after last spray.

The dissipation pattern of imidacloprid in mango fruits was studied at 1, 5, 10, 15 and 25 days after spraying. Data thus obtained are presented in Table 7.

**Table 7: Dissipation pattern of imidacloprid from 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		
3	0.00	50.00	100.00	-	-	5.0	10
6	0.00	37.50	75.00	100.00	-	7.5	15
SE $\pm$	-	4.52	3.76	-		-	-
CD at 1 %	-	11.38	NS	-		-	-
Significance		Sig.	NS				

N S: Not Significant

Per cent loss of imidacloprid residue at a dose of 3 ml/10 litre on 5<sup>th</sup> day was 50.00 which was reduced to 100 per cent on 10<sup>th</sup> day after spraying. The half-life of imidacloprid at a dose of 3 ml per 10 litre of water was found to be 5 days and waiting period was 10 days. The insecticide imidacloprid at a dose of 6 ml per 10 litre dissipated 37.50 per cent on 5<sup>th</sup> day then it was reduced to 75.0 per cent at 10<sup>th</sup> day after spraying and later on it was totally lost on 15<sup>th</sup> day. The half-life of imidacloprid at a dose of 6 ml per 10 litre was recorded to be 7.5 days and waiting period was 15 days.

The results of the present findings are relatively similar with the results of Mohapatra *et al.* (2011). They reported that imidacloprid residue degraded with the half-life of 3.06 and 4.16 days, at single and double doses respectively and persisted for 10 days.

#### **4.1.4 Monitoring Thiamethoxam residue in Mango fruits**

Thiamethoxam was sprayed on mango trees, the mango fruits were harvested and residue in fruits was estimated at 1,5,10,15 and 25 days after spraying using HPLC. The data pertaining to residue of thiamethoxam are presented in Table 8.

**Table 8: Residue of thiamethoxam in mango fruits at periodic interval**

<b>Dose g/10 lit</b>	<b>Average residue (ppm) at periodic interval (days)</b>				
	<b>1</b>	<b>5</b>	<b>10</b>	<b>15</b>	<b>25</b>
1	1.21	0.53	0.25	0.13	0.06
2	2.02	1.02	0.42	0.20	0.12
SE ±	0.30	0.12	0.07	0.06	0.05
CD at 1 %	0.70	0.29	0.16	NS	NS
Significance	Sig.	Sig.	Sig.	NS	NS



N S: Not Significant

Data pertaining to residue of thiamethoxam revealed that the recommended dose 1 g per 10 litre of water was significantly superior over double than recommended dose of 2 g per litre of water at 1, 5 and 10 days after spraying whereas, data was non-significant at 15 and 25 days after spraying.

The residue of thiamethoxam at a single dose of 1 g per 10 litre of water at one day after spraying was 1.21 ppm which was reduced to 0.53, 0.25, 0.13 and 0.06 at 5,10,15 and 25 days respectively. The residue of thiamethoxam at double than recommended dose at 2 g per 10 litre of water at one day was 2.02 ppm which was reduced to the extent of 1.02, 0.42, 0.20 and 0.12 ppm at 5,10,15 and 25 days after spraying.

Dissipation pattern of thiamethoxam in Mango fruits was studied at 1,5,10,15 and 25 days after spraying and data thus obtained are presented in Table 9.

Thiamethoxam at a dose of 1 g per 10 litre was dissipated to the extent of 56.19 per cent at five days after spraying and then it was reduced to 79.33, 89.25 and 95.04 per cent at 10, 15 and 25 days respectively. Whereas, double than recommended dose of thiamethoxam was reduced from 49.50, 79.20, 90.09 and 94.05 per cent at 5,10,15 and 25 days after spraying respectively. The half-life of thiamethoxam at a recommended dose of was 4.80 days and waiting period was 20 days. The half-life and waiting period of double than recommended dose of thiamethoxam was reported to be 5.20 and 20 days respectively. This indicated that the double dose of thiamethoxam is not advisable for the farmers as far as the cost and effect of the insecticide is concerned. The reports are in conformity with the results of Bhattacharjee and Dikshit (2016) wherein they

reported that thiamethoxam dissipated in mango from 1.93 and 3.71 mg kg<sup>-1</sup> after 2 h of spraying to 0.08 and 0.13 mg kg<sup>-1</sup> after 20 days of spraying at single and double doses, respectively. Its residue did not persist beyond 20 days in fruit.

**Table 9: Dissipation pattern of Thiamethoxam from mango fruits**

Doseg/10 lit	Percent loss of residue at periodic interval (days)					Half-life (days)	Waiting period (days)
	1	5	10	15	25		
1	0	56.19	79.33	89.25	95.04	4.80	20 days
2	0	49.50	79.20	90.09	94.05	5.20	20 days
SE ±		1.03	2.38	5.36	8.44		
CD at 1 %		2.80	NS	NS	NS		
Significance		Sig.	NS	NS	NS		

N S: Not Significant

#### 4.1.5 Monitoring Dimethoate residue in mango fruits

The residue of dimethoate at a recommended dose of 10 ml per 10 litre of water at one day after spraying was 1.92 ppm which was reduced to 0.57 and 0.12 ppm on 5 and 10 days after spraying respectively. The residue of dimethoate was not detectable after 15 days. Whereas, the residue of dimethoate at double than recommended dose of 20 ml per 10 ml per 10 litre was 3.58 ppm at one day after spraying which was reduced to 1.04 and 0.19 ppm at 5 and 10 days respectively. The insecticide dimethoate was not detectable after 15 days after spraying in mango fruits (Table 10).

**Table10: Residue of Dimethoate in mango fruits at periodic interval**

Dose ml/ 10 lit	Average residue (ppm) at periodic interval (days)				
	1	5	10	15	25
10	1.92	0.57	0.12	ND	ND
20	3.58	1.04	0.19	ND	ND
SE ±	0.53	0.05	0.06		
CD at 1 %	1.26	NS	NS		
Significance	Sig.	NS	NS		

N S: Not Significant

The results of the present findings are relatively similar with Bhattacharjee and Dikshit (2016) wherein they reported that dimethoate dissipated in mango from 2.81 and 5.34 mg kg<sup>-1</sup> after 2 h of application to 0.12 and 0.19 mg kg<sup>-1</sup> after 10 days of application at single and double doses, respectively. No residue was detected in fruit beyond 10 days after its application.

The dissipation pattern of dimethoate was studied under present investigation and presented in Table 11.

**Table 11: Dissipation pattern of Dimethoate from 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		
10	0.0	70.31	94.16	100	-	3.68	11
20	0.0	70.94	94.69	100	-	3.69	12
SE ±	-	0.04	0.06	-			
CD at 1 %	-	NS	NS	-			
Significance		NS	NS	-			

N S: Not Significant

Dimethoate at a dose of 10 ml per 10 litre of water was lost to the extent of 70.31 per cent which was then lost to 94.16 and 100 per cent at 10 and 15 days after spraying. Whereas, at double than recommended dose of 20 ml per 10 litre of water was lost from 70.94, 94.69 and 100 per cent at 5, 10 and 15 days after spraying. The half-life of dimethoate at single and double dose was 3.68 and 3.69 days whereas, waiting period was 11 and 12 days respectively. This indicated that the residue of dimethoate lasts up to 15 days. The double dose of dimethoate also leaves residue for 15 days only which is not advisable.

Bhattacharjee and Dikshit (2016) reported that dimethoate residue in mango degraded with the half-life of 2 days. These results are in agreement with the results of the present investigation.

## **2. To estimate the insecticidal residue from farmers orchards**

The mango fruits as per treatment were brought from farmers field for the estimation of insecticide residue. The residue of the insecticides deltamethrin, lambda cyhalothrin, imidacloprid, thiamethoxam and dimethoate was estimated by using HPLC at College of Forestry. The data thus obtained are presented in Table 12.

**Table 12: Residue of different insecticides in mango fruits of farmer's field**

Treatment	Average residue (ppm) one day after harvesting				
	Deltamethrin	Lambda cyhalothrin	Imidacloprid	Thiamethoxam	Dime-thoate
Farmers practice up to 5 sprays	ND	0.18	0.38	0.84	ND
Farmers practice up to 6 to 10 sprays	ND	0.44	0.40	0.18	0.02
Farmers practice above 10 sprays	ND	0.48	0.42	1.24	ND
Farmers practice with recommended sprays	ND	0.24	0.02	0.84	0.84
SE $\pm$	-	0.05	0.80	0.05	-
CD at 1 %	-	0.12	0.22	0.13	-
Significance	-	Sig.	Sig.	Sig.	-

ND: Not Detectable

The data on residue of the five insecticides under study in mango fruits of the farmers filed are presented in Table 12. Data revealed that the residue of Deltamethrin was not detectable. The reason might be the insecticide deltamethrin is generally sprayed at earlier stage of the crop and it does not leave residue for a longer time. The residue of lambda cyhalothrin was 0.24 ppm which uses University recommended schedule. The residue of the insecticide lambda cyhalothrin was 0.18, 0.44 and 0.48 ppm from the mango fruits of the farmersfield those who practice 5, 6-10 and more than 10 sprays respectively. The residue of imidacloprid from the mango fruits of the farmer's those use University recommended schedule was 0.02 ppm. The residue of imidacloprid was 0.38, 0.40 and 0.42 ppm those who use 5, 6-10 and more than 10 sprays respectively. The residue of thiamethoxam from the mango fruits of the farmers who use University schedule was 0.84 ppm. Whereas, the residue was

0.84, 0.18 and 1.24 ppm from the mango fruits of the farmers those practice up to 5 sprays, 6-10 sprays and more than 10 sprays. The residue of dimethoate from the mango fruits of the farmers field those practice University recommended schedule was 0.84 ppm. The farmers use up to 5 sprays, 6-10 sprays and more than 10 sprays recorded not detectable, 0.02 ppm and not detectable residue of dimethoate respectively. This indicated that the insecticide dimethoate is not used regularly by the farmers.

Unlike conventional insecticides belonging to organophosphates and carbonate group of compounds, synthetic pyrethroids are known to their photostability (Elliot *et al.* 1978) and rapidly undergoing conjugate formation to make glucosides at alcohol moieties in the chemical structures of plant constituents (Gaughan and Casida, 1978). As a result, their residues on mango fruit persisted for longer time. Further being non-systemic and highly lyophilic, they do not move from their site of application (Elliot, 1980).

## **CHAPTER V**

### **SUMMARY AND CONCLUSION**

The present investigation on “Estimation of insecticidal residue in Alphonso mango” was carried out in the Konkan region of Maharashtra during 2020. The summary of the results obtained during the present investigation are presented below.

The insecticides were sprayed at recommended and double than recommended doses on Mango trees. Mangoes from such treated trees were harvested at 1, 5, 10, 15 and 25<sup>th</sup> day after spraying. The collected mangoes were cut into pieces, blended, prepared extract of it, undergo clean up procedure and such clear sample was injected in High Performance Liquid Chromatography (HPLC) for residue estimation.

Data on residue of deltamethrin sprayed at recommended and double doses one day after spraying 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<sup>th</sup> day and thereafter was not detectable at 10, 15 and on 25<sup>th</sup> day.

The results of deltamethrin sprayed at double than recommended dose (18 ml/10 lit.) revealed that after one day, double recommended dose of deltamethrin (18 ml/10 lit.) recorded residue of 0.20 ppm which was reduced to 0.12 ppm on 5<sup>th</sup> day and thereafter was not detectable at 10, 15 and on 25<sup>th</sup> day.

Per cent loss of deltamethrin residue at a dose of 9 ml/10 litre on 5<sup>th</sup> 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<sup>th</sup> day and it was totally lost on 10<sup>th</sup> 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.

Data on residue of lambda cyhalothrin sprayed at recommended and double doses 1,5,10,15 and 25 days after spraying revealed that after one day, recommended dose of lambda cyhalothrin (6 ml/10 lit.) showed residue of 0.84 ppm which was reduced to 0.48, 0.21 and 0.04 ppm on 5, 10 and 15 days after spray. On 25<sup>th</sup> day the residue of lambda cyhalothrin was below detectable level.

The results of lambda cyhalothrin sprayed at double than recommended dose (12ml/10 lit.) revealed that after one day, double recommended dose of lambda cyhalothrin (12 ml/10 lit.) recorded residue of 1.40 ppm which was reduced to 0.95, 0.53 and 0.10 ppm on 5,10 and 15 days after spraying. The residue of lambda cyhalothrin at 25 days after spraying was below detectable level.

Per cent loss of lambda cyhalothrin residue at a dose of 6 ml/10 litre on 5<sup>th</sup> day was 42.83per cent which was reduced to 75.00 and 95.23 per cent at 10 and 15 days after spraying. The residue of lambda cyhalothrin was totally lost at 25 days after spraying. The half-life of lambda cyhalothrin at a dose of 6 ml per 10 litre was observed to be 8.12 days and waiting period was 18 days. The insecticide Lambda cyhalothrin at a dose of 12 ml per 10 litre dissipated 32.14 per cent on 5<sup>th</sup> day then it was reduced to 62.14 and 92.85 per cent at 10 and 15 days after spraying and later on it was totally lost on 25<sup>th</sup> day. The half-life of Lambda cyhalothrin at a dose of 12 ml per 10 litre was recorded to be 8.75 days and waiting period was 19 days.



Imidacloprid residue of the dose 3 ml per 10 litre in HPLC on day one after spraying was 0.12 ppm which reduced to 0.06 ppm at 5 days after spraying. It was below detectable level on 10<sup>th</sup> day after spraying. The residue of imidacloprid at double than recommended dose of 6 ml per 10 litre at one day after spraying was 0.16 ppm which was reduced to the extent of 0.10 and 0.04 ppm at 5 and 10 days after spraying and later on it was not detectable.

Per cent loss of imidacloprid residue at a dose of 3 ml/10 litre on 5<sup>th</sup> day was 50.00 which was reduced to 100 per cent on 10<sup>th</sup> day after spraying. The half-life of imidacloprid at a dose of 3 ml per 10 litre of water was found to be 5 days and waiting period was 10 days. Imidacloprid at a dose of 6 ml per 10 litre dissipated 37.50 per cent on 5<sup>th</sup> day then it was reduced to 75.0 per cent at 10<sup>th</sup> day after spraying and later on it was totally lost on 15<sup>th</sup> day. The half-life of imidacloprid at a dose of 6 ml per 10 litre was recorded to be 7.5 days and waiting period was 15 days.

The residue of thiamethoxam at a single dose of 1 g per 10 litre of water at one day after spraying was 1.21 ppm which was reduced to 0.53, 0.25, 0.13 and 0.06 at 5,10,15 and 25 days respectively. The residue of thiamethoxam at double than recommended dose at 2 g per 10 litre of water at one day was 2.02 ppm which was reduced to the extent of 1.02, 0.42, 0.20 and 0.12 ppm at 5,10,15 and 25 days after spraying.

Thiamethoxam at a dose of 1 g per 10 litre was dissipated to the extent of 56.19 per cent at five days after spraying and then it was reduced to 79.33, 89.25 and 95.04 per cent at 10, 15 and 25 days respectively. Whereas, double than recommended dose of thiamethoxam was reduced from 49.50, 79.20, 90.09

and 94.05 per cent at 5,10,15 and 25 days after spraying respectively. The half-life of thiamethoxam at a recommended dose of was 4.80 days and waiting period was 20 days. The half-life and waiting period of double than recommended dose of thiamethoxam was reported to be 5.20 and 20 days respectively.

The residue of dimethoate at a recommended dose of 10 ml per 10 litre of water at one day after spraying was 1.92 ppm which was reduced to 0.57 and 0.12 ppm on 5 and 10 days after spraying respectively. The residue of dimethoate was not detectable after 15 days. Whereas, the residue of dimethoate at double than recommended dose of 20 ml per 10 ml per 10 litre was 3.58 ppm at one day after spraying which was reduced to 1.04 and 0.19 ppm at 5 and 10 days respectively. The insecticide dimethoate was not detectable after 15 days after spraying in mango fruits.

The insecticide dimethoate at a dose of 10 ml per 10 litre of water was lost to the extent of 70.31 per cent which was then lost to 94.16 and 100 per cent at 10 and 15 days after spraying. Whereas, at double than recommended dose of 20 ml per 10 litre of water was lost from 70.94, 94.69 and 100 per cent at 5, 10 and 15 days after spraying. The half-life of dimethoate at single and double dose was 3.68 and 3.69 days whereas, waiting period was 11 and 12 days respectively.

The mango fruits as per treatment were brought from farmers field for the estimation of insecticide residue. The residue of the insecticides deltamethrin, lambda cyhalothrin, imidacloprid, thiamethoxam and dimethoate was estimated by using HPLC at College of Forestry.

The data on residue of the five insecticides under study in mango fruits of the farmers field revealed that the residue of deltamethrin was not detectable. The residue of lambda cyhalothrin was 0.24 ppm which uses University recommended schedule. The residue of the insecticide lambda cyhalothrin was 0.18, 0.44 and 0.48 ppm from the mango fruits of the farmers field those who practice 5, 6-10 and more than 10 sprays respectively. The residue of imidacloprid from the mango fruits of the farmer's those use University recommended schedule was 0.02 ppm. The residue of imidacloprid was 0.38, 0.40 and 0.42 ppm those who use 5, 6-10 and more than 10 sprays respectively. The residue of thiamethoxam from the mango fruits of the farmers who use University schedule was 0.84 ppm. Whereas, the residue was 0.84, 0.18 and 1.24 ppm from the mango fruits of the farmers those practice up to 5 sprays, 6-10 sprays and more than 10 sprays. The residue of dimethoate from the mango fruits of the farmers field those practice University recommended schedule was 0.84 ppm. The farmers use up to 5 sprays, 6-10 sprays and more than 10 sprays recorded not detectable, 0.02 ppm and not detectable residue of dimethoate respectively.

From the results of the present investigation it can be concluded that the insecticides deltamethrin, imidacloprid and dimethoate does not leave residue for a longer period but the insecticides lambda cyhalothrin and thiamethoxam leaves residue up to 15 and 25 days respectively, therefore they should not be used at a later part of the crop.

## LITERATURE CITED

- Akhtar S., Yaqub G., Hamid A., Afzal Z. and Asghar S. (2018). Determination of pesticide residues in selected vegetables and fruits from a local market of Lahore, Pakistan. *Curr. World Environ.*, **13**(2) 242-250.
- Alrahman, S.H. (2014). Residue and dissipation kinetics of thiomethaxam in a vegetable field ecosystem using QuEChERS methodology combined with HPLC-DAD. *Food Chemistry* **159**: 1-4.
- Anonymous, (2018). Horticultural Statistics at a Glance 2018, Gov. of India. pp: 146, 180.
- Anwar, R., Saeed, A., Muhammad, Y., Waqar, A. and N. Muhammad (2011). Bi-monthly nutrient application programme on calcareous soil improves flowering and fruit set in mango (*Mangifera indica* L.). *Pak. J. Bot.* **43**(2): 983-990.
- Arora, P.K., Jyoti, G., Randhawa, P., Singh, B., Battu, R.S. and Singh, B.A. (2008). Dissipation of imidacloprid on Kinnow mandarin fruits under sub-tropical conditions. *Indian J. Hort.* **65**(3): 277-279.
- Arora, S. (2008). Analysis of insecticides in okra and brinjal from IPM and non-IPM fields. *Environmental monitoring and assessment*. **151**. 311-5. Doi: 10.1007/s10661-008-0272-.
- Arora, S. A., Singh, America, Shukla, R. P. and Singh, J. (2006). Residues of chlorpyrifos and monocrotophos in IPM and non IPM mango orchards. *Pesticide Research Journal*, **18**(1): 76-78.

- Awasthi, M.D. (1988). Persistence of synthetic pyrethroids residues on mango fruits. *Acta Horticulture*. **231**: 612-616.
- Banerjee, T., Banerjee, D., Roy, S., Banerjee, H. and Pal, S. (2012). A Comparative study on the persistence of imidacloprid and beta-cyfluthrin in vegetables. *Bulletin of Environmental Contamination and Toxicol* **89**: 193-196.
- Bhattacharjee A. K. and Dikshit A. (2016), Dissipation kinetics and risk assessment of thiamethoxam and dimethoate in mango. *Environ Monit Assess* **188**: 165.
- Butani, D. K. (1974). Insect pests of fruit crops and their control (Mango). *Pesticides*. **8**(3): 37-41.
- Chandra, S., Kumar, M., Mahindrakar, A.N. and Shinde, L.P. (2014). Persistence pattern of chlorpyrifos, cypermethrin and monocrotophos in okra. *International Journal of Advanced Research* **2**(2): 738-743.
- Charan, P.D., Ali, S.F., Kachhawa, Yati and Sharma, K.C. (2010). Monitoring of Pesticide Residues in Farmgate Vegetables of Central Aravalli Region of Western India *American-Eurasian J. Agric. & Environ. Sci.*, **7**(3): 255-258. Fide: [http://idosi.org/aejaes/jaes7\(3\)/2.pdf](http://idosi.org/aejaes/jaes7(3)/2.pdf).
- Chauhan, R., Kumari, B., & Sharma, S. S. (2013). Persistence of thiamethoxam on okra fruits. *Pesticide Research Journal*, **25**, 163–165.
- Djouaka R., Soglo M. F., Michael OlugbengaKusimo, RazackAdéoti, Armand Talom, Francis Zeukeng, Armand Paraiso, Victor Afari-Sefa , May-GuriSaethre, Victor Manyong, ManueleTamo, Jeff Waage, Jo Lines and George Mahuku (2018). The Rapid Degradation of Lambda-Cyhalothrin Makes Treated Vegetables Relatively Safe for

Consumption. *Int. J. Environ. Res. Public Health*, **15**, 1536; doi:10.3390/ijerph15071536.

Dubale, J. J., Patil, P. D., Mule, R. S. and Jalgaonkar, V. N. (2010). Dissipation of cypermethrin in mango. *Pestology*. **34**(12): 30-32.

Dubale, J.J., Patil, P.D., Mule, R.S. and Jalgaonkar, V.N. (2011). Insecticide residues in alphonso mango fruits. *Pestology* **35**(1): 30-31. Fide:

[http://www.pakbs.org/pjbot/PDFs/43\(4\)/PJB43\(4\)1915](http://www.pakbs.org/pjbot/PDFs/43(4)/PJB43(4)1915).

Elliot M. (1980). Established pyrethroids. *Pestic. Sci.* **11**: 119-128.

Elliot M., Janes N. F. and Patter C. (1978). The future of pyrethroids in insect control. *A. Rev. Ent.* **23**: 443-469.

Gaughan L.C. and Casida J. E. (1978). Degradation of cis and transpermethrin on cotton and bean plants. *J. Agric. Food. Chem.* **26**: 526-529.

Gupta, A., Singh, B., Parihar, N.S. and Bhatnagar, A. (1998). Pesticide residue in the farm gate samples of bottle gourd, cauliflower, cabbage and fenugreek at Jaipur. *Pesticide Research Journal*. **10**: 86-90.

Gupta, S. P., Singh, S. P., Satyanarayana, P. and Kumar, N. (2015). Dissipation and decontamination of imidacloprid and lambda - cyhalothrin residues in brinjal. *Internat. J. Plant Protec.*, **8**(2): 379-383.

Hafeez, A. and Rizvi, S.M.A. (1993). Residues of some synthetic pyrethroids and monocrotophos in/on okra fruits. *Indian J. Pl. Prot.* **21**(1): 44-46.

- Hafez, O. H. and Singh, B. (2016). Persistence of thiomethaxam in/on tomato fruits and soil. *Journal of insect Science* **29**(1): 25-31.
- Handa, S. K., Agnihotri, N. P. and Kulshrestha, G. (1999). Pesticide residues: significance, management and analysis, *Research Periodicals and book publishing home*, Texas, USA pp: 56-59.
- Hassan, M., Ahmad, F., Sagheer, M., Iqbal, M.F. and Tariq, M. (2005). Residual persistence of chlorpyrifos, imidacloprid and acephate in brinjal fruits. *Pak. Entomol.* **27**(1): 53-55.
- Hem, L., Park, J. H., & Shim, J. H. (2010). Residual analysis of insecticides (lambda-cyhalothrin, lufenuron, thiamethoxam and clothianidin) in pomegranate using GC- $\mu$ ECD or HPLC-UVD. *Korean Journal of Environmental Agriculture*, **29**, 257 –265.
- Jallow M.F., Awadh D.G., Albaho M.S., Devi V.Y., Ahmad N. (2017). Monitoring of Pesticide Residues in Commonly Used Fruits and Vegetables in Kuwait. *International Journal of Environmental Research and Public Health.*; **14**(8):833.
- Kadam, D.R., Kale, V.D., Deore, B.V., Bade, B.A. and Jadhav, R.S. (2012). Residues and dissipation of imidacloprid (Confidor 17.8 SL) in pomegranate fruits. *Pestology* **35**(3): 29-33.
- Karmakar, R., & Kulshrestha, G. (2009). Persistence, metabolism and safety evaluation of thiamethoxam in tomato crop. *Pest Management Science*, **65**, 931 –937.
- Kumari B., Madan, V. K., Kumar, R. and Kathpal, T. S. (2002). Monitoring of seasonal vegetables for pesticide

residues. *Environmental Monitoring and Assessment*, **74**: 263-270.

Mahdavian, E. S. and Somashekhar, R. K. (2010). Synthetic pyrethroids multiresidues in grapes from Southern India. *Journal of Sciences and Technology*, **6**(2): 104-110.

Mahapatra, S. (2014). Residue dynamics of chlorpyrifos and cypermethrin in/on pomegranate (*Punicagranatum* L.) fruits and soil. *International Journal of Environmental and Analytical Chemistry* **94**: 14-15.

Mohapatra S, Deepa M, Jagadish G. K. (2011). Residues of beta cyfluthrin and imidacloprid in/on mango (*Mangifera indica* L.). *Bull Environ Contam Toxicol* **87**: 202-207.

Mohapatra, S. and Ahuja, A. K. (2010). Persistence and dissipation of lambda cyhalothrin in/on mango (*Mangifera indica* L.). *The Indian Journal of Agricultural Sciences*, **80** (4): 306-308.

Mohapatra, S., Deepa, M., Lekha, S., Nethravathi, B., Radhika, B. and Gourishanker, S. (2012). Residue dynamics of spirotetramat and imidacloprid in/on mango and soil. *Bulletin of Environmental Contamination and Toxicology*, **89** (4): 462-46.

Mukherjee, I., Singh, S., Sharma, P.K., Jaya, M. and Kulshrestha, G. (2007). Extraction of multi-class pesticide residues in mango fruits: application of pesticides residues in monitoring of mangoes. *Bulletin of Environmental Contamination and Toxicology*, **78**: 380-383.

Munj, A. Y. (2011). Determination of Pre Harvest Interval (PHI) of commonly use insecticides and fungicides in Alphonso mango. RRC report of Department of Agricultural



Entomology, College of Agriculture, Dr. B.S.K.K.V., Dapoli  
p.12.

Okediran, O., Dauda, M. S., &Kolawole, S. A. (2019). Assessment of Pesticide Residues in Fresh Vegetables from Three Major Markets in Lagos Using QuEChERS Method and GC-MS. *International Research Journal of Pure and Applied Chemistry*, **19** (4), 1-8.

<https://doi.org/10.9734/irjpac/2019/v19i430120>

Pandit, G. &Gharde, S. &Chowdhury, N. &Ghosh, J.. (2016). Dissipation of Imidacloprid Residues in Okra Leaves, Fruits and Soil in Northern Region of West Bengal. **28**, 20-24.

Parveen Z., Riazuddin, I. S., Khuhro, M. I. and Bhotto, M. A. (2011). Monitoring of multiple pesticide residues in some fruits in Karachi, Pakistan. *Pakistan Journal of Botany*, **43** (4): 1915-1918.

Phartial T. and Srivastava R.M. (2014).Dissipation of imidacloprid on lemon fruit rind under tarai agro-climatic condition of Uttarakhand. *J. ent. Res.*, **38**(4): 285-288 pp. 91,96, 281.

Pujeri, U. S., Yadawe, M. S., Hiremath, S. C. and Pujar, A. S. (2010).Monitoring pesticide residues in pomegranate of Bijapur district Karnataka. *Journal of Pharmaceutical and Biomedical Sciences*, **2**(11):1-2. Fide: [www.jpbums.info/index.php?option=com\\_docman&task=doc](http://www.jpbums.info/index.php?option=com_docman&task=doc).

Rabea M. M., Ibrahim E. S., Elhafny D. and Bayoumi M. A.. 2018. Determination of Dinotefuran and Thiamethoxam Residues in Pepper Fruits under Greenhouse Conditions

using the QuEChERS Method and HPLC/DAD. *Egypt. J. Chem.* Vol. **61**, No. 2, pp. 249 – 257.

Ramadan G., Shawir M., El-bakary A. and Abdelgaleil S. (2016). Dissipation of four insecticides in tomato fruit using high performance liquid chromatography and QuEChERS methodology. *Chilean Journal of Agricultural Research* **76**(1): 129-133.

Reddy, N. C., Anitha, K. D., Lakshami, B. K. M. and Reddy, J. (2013). Residue dynamics of imidacloprid and hexaconazole on mango. *International Journal of Bio-resources and Stress Management*, **4** (2):263-265. Fide:<http://www.indianjournals.com/ijor.aspx?target=ijor:ijbsm&volume=4&issue=2&article=028&type=pdf>.

Salvi, B. R., Haldankar, P.M., Rane, A.J. and Burondkar, M.M.(2018). Area and production of mango, *Advances in mango production technology*. Dr B.S.K.K.V., Dapoli. pp 4.

Sen, S. and Chowdhury, A. (1999). Long term residue and persistence study of deltamethrin in brinjal. *Pestology***23**(9): 35-38.

Shah, A. H., Patel, G. M. and Jhala, R. C. (1973). Field evaluation of insecticides against mango hopper, *Amritodus atkinsoni* (Leth.). *Pestology*, **8** (3): 26.

Sharma, P. C., Chandresh, P. and Sharma, S. (2018). Persistence of Imidacloprid, Indoxacarb and Lambda-Cyhalothrin on Tomato (*Solanum lycopersicum* L.) Under Protected Cultivation. *Int. J. Curr. Microbiol. App. Sci.* **7**(7): 2783-2794. Doi:<https://doi.org/10.20546/ijcmas.2018.707.325>.

- Shinde, L.P., Kolhatkar, D.G., Baig, MMV. and Subhash Chandra (2012). Study of cypermethrin residue in okra leaves and fruits. *IJRPC***2**(2).
- Shrivastava, K. P. (1988). Techniques in pesticide residue analysis. *A Text Book of Applied Entomology*, Kalyani Publishers: 294-300.
- Singh, Y., Mandal, K. and Singh, B. (2015). Persistence and risk assessment of cypermethrin residues on chilli (*Capsicum annum* L.). *Environmental Monitoring Assessment***187**:120.
- Suarez-Jacobo, Angela & Alcantar, Victor & Alonso, D & Heras-Ramírez, M & Elizarragaz de la Rosa, Dalaú & Lugo, Yadira & Gaspar, Octavio (2017). Pesticide residues in orange fruit from citrus orchards in Nuevo Leon State, Mexico. *Food additives & contaminants. Part B, Surveillance*. Doi: 10.1080/19393210.2017.1315743.
- Tahir S, Anwar T, Ahmad I, Aziz S, Mohammad A, Ahad K. (2001). Determination of pesticide residues in fruits and vegetables in Islamabad market. *Journal of Environment Biology*.**22**(1):71-74.
- Tahir, U. M., Naik, I. S., Rehman, S. and Shahzad, M. (2009). A quantitative analysis for the toxic pesticides residues in marketed fruits and vegetables in Lahore, Pakistan. *Biomedica*, **25**: 171-174.
- Vijayalakshmi, K. (2002). Dissipation of quinalphos on mango (*Mangifera indica* L.) fruit. *Tropical Agriculture*, (Trinidad). **78** (1): 39-42.
- Xiuguo W. & Xiang, Zhenbo & Yan, Xiao yang & Sun, Huiqing & Li, Yiqiang. (2013). Dissipation Rate and Residual Fate of Thiamethoxam in Tobacco Leaves and Soil Exposed to Field

Treatments. *Bulletin of environmental contamination and toxicology*. **91** (2). Doi: 10.1007/s00128-013-1043-2.



**Plate I: Application of pesticidal treatments**



**Plate II: High Performance Liquid Chromatograph equipped with PDA**



mango sample Homogenizing sample

Adding of acetonitrile Homogenized sample





Blending Vacuum extracted sample

### **Plate III: Extraction and clean-up steps for residue analysis**

Cont...



Sample concentration  
 $\text{Na}_2\text{SO}_4$   
 with DCM

Dried over

