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DIST.RATNAGIRI, MAHARASHTRA

Title of Thesis : Estimation of Insecticidal Residue in Alphonso Mango Fruits.

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Year : 2015-16

Regd. No. : ADPM/15/2434

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ABSTRACT

Mango (*Mangifera indica* L.) is most important commercial crop of India. In India, Maharashtra state ranks 8th in production of mango. The injudicious and indiscriminate application of insecticides to crops result in residues in food and food commodities with consequential hazards. The delicious fruit mango 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 are not only for quality of agricultural produce but also the safety and environmental friendly production. To cope with the contemporary international market there is dire need to carry out systemic research on pesticide residue in mango fruits. Glance through the hazardous of pesticide residues, a research programme was premediated as “Estimation of Insecticidal Residue in Alphonso Mango Fruits”.

In order to find out insecticidal residue in Alphonso mango fruits, two orchards were selected. One orchard is located at department of Agronomy, College of Agriculture, Dapoli, Dist. Ratnagiri (orchard 1) and another orchard is belongs to the private farmer Mr. Shrikrushna Phatak, Jalgaon (Dapoli) Dist. Ratnagiri (orchard 2). From each mango orchard, 30 mango trees were selected for the experiment. From orchard one where insecticidal application schedule

recommended by Dr.B.S.K.K.V., Dapoli was followed, mango fruit samples at egg stage as well as at harvest stage were collected to study insecticidal residue and their persistence. From orchard two mango fruit samples at harvest stage were collected to study insecticidal residue.

It is evident from the results that cypermethrin and thiomethaxam were used in higher concentration in orchard two as compared to orchard one. In spite of having higher dose, cypermethrin residues were found below detectable limit of 0.2 ppm in both cases i.e. 0.05 ppm for orchard one and 0.05 ppm for orchard two. While thiomethaxam residues were not detected in any fruit samples from both mango orchards. Thus the insecticidal spray schedule, recommended for management of pests infesting mango blossom by Dr.B.S.K.K.V., Dapoli as well as insecticidal spray schedule followed by private farmer are safe from pesticide residue point of view.

The study of total nutrient content of mango fruit samples from both orchards revealed that the total N, P, Ca, Mg, S and micronutrients viz. Fe, Mn, Zn, Cu were higher in orchard one than orchard two except total K.

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CHAPTER I

INTRODUCTION

Mango (*Mangifera indica* L.) belonging to family Anacardiaceae is the leading fruit crop of India and is believed to have originated from south east Asia. Besides delicious taste, excellent flavour and attractive fragrance, it is rich in vitamin A and C. It is known as “the King of the fruits”.

India is the major producer of mango in the world. In the year 2014-15, the 2217.0 thousand ha area of India was covered with mango, it produced 18506.0 thousand tonnes of mangoes. In India, Maharashtra state ranks 8th in production of mango, which occupies the maximum area of 155.97 thousand ha, with the production of 868.60 thousand tonnes. The average productivity of mango in India was around 8.3 tonnes per ha⁻¹ (Anonymous, 2015).

The Konkan region is famous and well known for mango production with an area of about 0.14 million ha with the production of 0.29 million tons and 2.07 tonnes ha⁻¹ productivity. Particularly, the two districts of the region viz. Ratnagiri and Sindhudurg are known as ‘Mango baskets’. Of these, the area under mango production in Ratnagiri district is approximately 0.063 million ha with production of about 0.12 million tonnes having a productivity of 1.9 tonnes ha⁻¹, respectively (Anonymous, 2014).

Damage due to insect pest is one of the limiting factor for low yield of mango fruits. It is reported to be infested by as many as 400 insects and non-insect pest in the world, of which 188 species are found infesting mango in India (Tandon and Shrivastava, 1982). The common insect pests are mango leafhopper, mealy bug, leaf webber and fruit fly. The major loss of about 60 % is due to leafhopper and mango hopper and chemical control is by far the only method presently used in its quick management (Mukherjee *et al.*, 2007)

The injudicious and indiscriminate application of insecticides to crops result in residues in food and food commodities with consequential hazards. The extent of hazard depends on the amount of insecticide residue on crops and their toxicity. Since most insecticides are toxic in nature, their continuous ingestion by man even

in trace amounts, can result in accumulation in body tissues with serious adverse effects on health (Handa *et al.*, 1999). According to Environmental Protection Agency (EPA); 60 percent herbicides, 90 percent of fungicides and 30 percent insecticides are known to be carcinogenic (cancer causing).

Many insecticides being highly stable continue to kill insect, long after their application. This ability of insecticide is called as “residual action” which is both advantageous and disadvantageous. Advantageous because a single application achieve more kill, spread 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 parasites, predators and pollinators (non-target) also run the risk of being killed due to prolonged residual action of toxicants (Shrivastava, 1988). The disadvantages of insecticide use known as 4Rs (Resistance, Resurgence, Risk and Residue), are well known. Insecticidal residues are also becoming a major obstacle in reducing India’s export to international market. 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 pest. To minimize the economic losses caused by these noxious pest, various pesticides are being used over the mango crop on massive scale. Due to lack of awareness, the farmers of our country do not follow the prescribed dosage and use pesticides at any stage of crop which results in accumulation of residue in fruits.

For the management of mango hoppers, Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli has recommended a six spray schedule of insecticides. 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 pollute the fruits and be harmful for human health. Therefore, pesticide residue is becoming a major food safety concern of consumers and government. So, in order to remove residual effect of insecticides, which are toxic, we should know the exact dose which should be recommended to the farmers.

The delicious Alphonso mango fruits are highly appreciated in many countries. Thus it opens tremendous opportunities for its export and fetch premium price in the world markets. Today market demands are not only for quality of agricultural produce but also the safety and environmental friendly production. To cope with the contemporary international market there is dire need to carry out systemic research on pesticide residue in mango fruits. Glance through the hazardous of pesticide residues, a research programme was premediated as 'Estimation of Insecticidal Residue in Alphonso Mango Fruits' with the following objectives:

1. To find out the insecticidal residues in Alphonso mango fruits after the application of recommended schedule of insecticides.
2. To find out the persistence of insecticides in Alphonso mango Fruits.
3. To compare the insecticidal residue levels in Alphonso mango fruits from orchard using recommended schedule and orchard following their own schedule.
4. To analyse the nutrient content in fruits.

(N, P, K, Ca, Mg, S, Fe, Mn, Zn, Cu).

CHAPTER II

REVIEW OF LITERATURE

The present investigation was carried out in the Konkan region of Maharashtra in lateritic soils to study the “Estimation of insecticidal residue in Alphonso mango fruits” in the year 2016. The available literature pertaining to the present investigation have been reviewed and presented under the following suitable headings.

2.1 Insecticidal residues in fruits and vegetables.

2.2 Persistence of insecticides in fruits and vegetables.

2.3 Nutrient content in Mango fruits (N, P, K, Ca, Mg, S, Fe, Mn, Zn, Cu).

As no much literature is available on insecticidal residue in Alphonso mango fruits, the related studies in respect of cypermethrin, quinolphos, imidacloprid, thiomethaxam residues have been reviewed and presented as follows.

2.1 Insecticidal residue in fruits and vegetables

2.1.1 Cypermethrin

Gajbhiye *et al.* (1985) studied the efficacy and residues of some newer synthetic pyrethroids in okra. Cypermethrin, permethrin and fenvalerate were applied at 75, 150 and 200 g a.i. ha⁻¹. While deltamethrin, fluvalinate, flucythrinate and fenpropathrin were applied at 25 g a.i. ha⁻¹. The initial deposits of cypermethrin, permethrin and fenvalerate were 0.25, 0.75 and 0.83 ppm respectively. The relative deposits of deltamethrin, flucythrinate, fluvalinate and fenpropathrin ranged from 0.06 and 0.10 ppm only. The residues of all the insecticides quickly dissipated with time and reached below 0.05 ppm on 5th day and became non detectable on 10th day except fenvalerate. The fenvalerate was found to leave 0.01 ppm residue on 10th day. The residue of permethrin, cypermethrin reached below MRL in two days and fenvalerate in five days. Since residue of flucythrinate, fluvalinate, fenpropathrin and deltamethrin were less than 0.05 ppm on 2nd day. Thus, three day waiting period can be considered as safe for consumption.

Awasthi (1989) studied dissipation of synthetic pyrethroids viz., permethrin, cypermethrin, fenvalerate, deltamethrin and monocrotophos residues on acid lime fruits. The pyrethroids dissipated with half-life of 2.0 to 4.0 days from their minimum effective and higher application rates. Based on the dissipation pattern of residues from different treatments in relation to respective MRL, the waiting period of 8-13 days for permethrin, 0-1 day for cypermethrin, 1-4 days for fenvalerate and 0-2 days for deltamethrin compared with 14-15 days for monocrotophos was suggested.

Singh and Kalra (1992) determined residues of cypermethrin in brinjal fruits, leaves and soil at concentration of 50 and 100 g a.i. ha⁻¹. The samples of fruits, leaves and soil were collected at 0, 1, 2, 5 and 10 days interval after 6th and 8th spray and analysed by gas liquid chromatography. They reported that, during 1982 the mean initial deposit of cypermethrin on fruit was 0.35 mg kg⁻¹ after the 6th spray at 50 g a.i. ha⁻¹ which declined to 14, 48 and 85 per cent in 1, 2 and 5 days, respectively. The application of 100 g a.i. ha⁻¹ resulted in initial deposit of 0.62 mg kg⁻¹ which declined to 0.48, 0.32, 0.10 and 0.05 mg kg⁻¹ after 1, 2, 5 and 10 days, respectively. The mean initial deposit of cypermethrin in the fruits after 8th spray at 50 g a.i. ha⁻¹ was 0.73 mg kg⁻¹ which dissipated by 36 per cent in two days. Residue level at the end of 10 day was 0.08 mg kg⁻¹ with 89 per cent dissipation of residue. At higher dose of 100 g a.i. ha⁻¹, the mean initial deposit of 1.43 mg kg⁻¹ dissipated to 1.26, 0.91, 0.23 and 0.07 mg kg⁻¹ after 1, 2, 5 and 10 days of spraying, respectively. During 1983, almost similar rate of dissipation was observed. Half-life values on brinjal fruits varied between 2.2 and 2.9 days for cypermethrin.

Awasthi (1993) reported insecticide residues on mango fruits, resulting from plant protection sprays, were reduced to 66-68 per cent for dimethoate and fenthion as against 21-27 per cent for fenvalerate and cypermethrin simply by washing. The study also reported that the peeling off the fruits pericarp was found to dislodge 100 per cent of residues in all cases.

Pawar and Jadhav (1993) conducted field experiment to study dissipation of lambda cyhalothrin, chinmax, cypermethrin, fenvalerate and endosulfan in okra.

Three sprays of insecticides were given at an interval of 15 days. After third spraying marketable size okra fruits were collected for dissipation on 0,1,3,5,7,10 and 14 days. In lambda cyhalothrin waiting period of 3.1 to 3.3 days in fruits were recorded. The waiting period in chinmax and cypermethrin ranged from 4.2 to 4.8 and 3.49 days respectively in fruits. Fenvalerate residues were below MRL after 7.32 days. Endosulfan waiting period were 5.79 and 5.53 days.

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⁻¹), cypermethrin (60 g a.i. ha⁻¹), permethrin (120 g a.i. ha⁻¹), fenvalerate (120 g a.i. ha⁻¹) and monocrotophos (320 g a.i. ha⁻¹) on green fruits of okra were 0.02 – 0.41 ppm on the 2nd day, and 0.01- 0.15 ppm on the 5th 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.

Islam *et al.* (2009) carried out an experiment to study residues of diazinon, malathion, chlorpyrifos and cypermethrin in cauliflower. All the insecticides were applied at recommended and double recommended dose and reported that, the amount of residues of diazinon at the recommended dose was 1.085 mg kg⁻¹ and at double of the recommended dose was 1.64 mg kg⁻¹. Similarly, chlorpyrifos present at recommended dose was 1.62 mg kg⁻¹ and at double recommended dose was 2.243 mg kg⁻¹. Hence, diazinon and chlorpyrifos both were detected above MRL (0.05 mg kg⁻¹). Malathion and cypermethrin were not detected in samples.

Dubale *et 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.

Munj (2011) carried out an experiment to determine pre harvest interval of cypermethrin in Alphonso mango fruits. Cypermethrin was applied at the spray concentration of 0.0075 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 cypermethrin residue at 42 days after spray.

Dubale *et al.* (2011) carried out field experiment on Alphonso mango fruits to study the residues of cypermethrin, endosulfan, imidacloprid, methyl demeton, quinolphos 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.

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 17th 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 21th day at concentration of 100 ppm.

.Chandra *et al.* (2014 a) 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⁻¹. 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⁻¹ respectively. The residues of pesticides reached below detection in the 15, 17 and 19 days for chlorpyrifos, cypermethrin and monocrotophos respectively.

2.1.2 Quinolphos

Dethe *et al.* (1997) studied residues of lindane and quinolphos in green chillies. The initial residues of lindane was 1.42 to 2.10 ppm at the dose 150 to 300

g a.i. ha⁻¹, which were less than MRL of 3 ppm prescribed for lindane. With similar treatments of quinolphos, initial residues of 1.23 to 1.98 ppm dissipated to MRL of 0.25 ppm in 4.14 to 4.85 days. Hence, waiting period of 1 day for lindane and 5 days for quinolphos were suggested.

Kale *et al.* (1997) studied dissipation of quinolphos on brinjal fruits. The initial deposits of 1.60 to 2.15 ppm with concentration of 500 to 1000 g a.i. ha⁻¹ dissipated to MRL of 0.25 ppm within 3.16 to 4.72 days. The residual half-life was 1.10 to 1.37 days. Hence, waiting periods of 5 days were suggested.

Mathew *et al.* (1998) conducted field experiment to study the dissipation of quinolphos and monocrotophos residues in cardamom capsules. The residues of quinolphos reached below the tolerance limit of 0.25 ppm within 23 to 24 days in fresh cardamom and with 20 to 21 days in cured cardamom. The periods for monocrotophos did not differ between fresh and cured cardamom (22 to 23 days) because of systemic nature of the residues.

Geeta and Ragupathy (1999) studied harvest time residues of quinolphos 20 AF in cashew fruits and nuts. The residue level of quinolphos 20 AF applied 4 times at monthly intervals at 0.5, 0.2 and 0.1 per cent were below detectable limits of 0.05 and 0.1 ppm in both the fruits and nuts, respectively at harvest time.

Kang *et al.* (1999) carried out field experiment to study residues of monocrotophos and quinolphos in different fractions of paddy. Monocrotophos was applied at 500 and 1000 g a.i. ha⁻¹ concentration while quinolphos was applied at 250 and 500 g a.i. ha⁻¹ concentration. The residues of monocrotophos were found to be 2.404 and 5.904 mg kg⁻¹ at recommended and double recommended dose respectively. Similarly, the residues of quinolphos were not detected in rice grain and rice bran samples at recommended and double recommended dose.

Vijayalakshmi (2002) studied dissipation of quinolphos on mango fruits. Quinolphos was sprayed at 15 days intervals at the rate of 0.05 and 0.1 per cent on mango fruits at marble size stage. The average initial deposits at 0.05 per cent concentration was 3.76 ppm which dissipated to 0.14 ppm with 96 per cent dissipation after 15 days. The average initial deposits at 0.1 per cent concentration

was 6.75 ppm which reduced to 0.23 ppm, with 97 per cent dissipation after 15 days. Hence, she reported a waiting periods of 11.5 and 14.2 days after treatments for the safe consumption of fruits at 0.05 and 0.1 per cent dose, respectively before harvest.

Munj (2011) carried out an experiment to determine pre harvest interval of quinolphos in Alphonso mango fruits. Quinolphos was applied at the spray concentration of 0.05 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 quinolphos residue at 42 days after spray.

Dubale *et al.* (2011) carried out field experiment on Alphonso mango fruits to study the residues of cypermethrin, endosulfan, imidacloprid, methyl demeton, quinolphos 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 insecticide residue and hence safe for human consumption.

2.1.3 Imidacloprid

Hule (1998) carried out field experiment to study the residues of imidacloprid in green chillies. The results revealed that, the spraying of 0.01 per cent imidacloprid (200 SL) did not leave detectable levels of residues in green chillies harvested at 30 days after 3rd spray.

Sharma and Awasthi (1998) studied persistence pattern of imidacloprid in mango fruits. Imidacloprid was sprayed on mango trees at the panicle emergence stage and repeated 21 days later at 0.4, 0.8 and 1.2 ml L⁻¹. Mango fruits picked at the stone formation stage contained 0.209, 0.389 and 0.563 ppm imidacloprid residues which dissipated to 0.156, 0.274 and 0.311 ppm, respectively, at maturity.

Whole fruit samples as well as fruit pulp at harvest, however, did not show any detectable residues of imidacloprid.

Indumathi *et al.* (2001) studied uptake and dissipation of imidacloprid residues in okra. Uptake study was carried out by treating the seed with imidacloprid (Gaucho 70 WS) at 9 g a.i. kg⁻¹ of seeds. The residues persisted in plant for more than 30 days after germination and could not be detected in fruits harvested at 50, 55 and 60 days after germination. The dissipation study was carried out by spraying okra crop at fruiting stage with Confidor 200 SL at 0.3 and 0.6 ml L⁻¹. The half-life values ranged from 2-4 days. The residues, however, become non-detectable within 10 days at lower concentration and 15 days at higher concentration.

Mukherjee and Gopal (2001) conducted field experiment to estimate residues of imidacloprid by using its new liquid formulation as seed dressing in cotton. Cotton seeds were treated with imidacloprid (Gaucho 600 FS) at recommended dose of 6, 9 and 12 ml kg⁻¹ and two high dosages i.e. 18 and 24 mg kg⁻¹. The residues of imidacloprid were not detected in cotton lint and seed. The residues were invariably below detectable level i.e. 0.05 mg kg⁻¹ following the seed dressing at all the five different dosages at 6-24 ml kg⁻¹.

Kharbade *et al.* (2003) studied residues of imidacloprid on chilli. Three sprays of imidacloprid 17.8 per cent SL was evaluated at 100, 150 and 300 ml ha⁻¹ at an interval of 15 days by initiating first at 7 weeks after transplanting. The initial deposits after third spray at 100 and 150 ml were 0.38 to 0.58 ppm which dissipated to BDL of 0.05 ppm in 4.19 to 5.48 days, respectively. At higher dose of 300 ml ha⁻¹, the initial residues of 1.21 ppm dissipated to BDL in 8.16 days. The residue half-life was 1.47 to 1.78 days. However, a period of 5 to 6 days was reported as safe waiting periods.

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 hrs after spraying, were maximum 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 hrs 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 hrs 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.* (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 kg⁻¹ respectively. The corresponding values in the pulp were observed to be 0.03 and 0.04 mg kg⁻¹, respectively. A sudden decline in the residue levels in the rind and a corresponding increase in the pulp was observed on 5th 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.

Dubale *et al.* (2011) carried out field experiment on Alphonso mango fruits to study the residues of cypermethrin, endosulfan, imidacloprid, methyl demeton, quinolphos 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 insecticide residue and hence safe for human consumption.

Diwan *et al.* (2012) studied dissipation of β -cyfluthrin and imidacloprid as combination product in/on mango. Three sprays of ready mix formulations; β -cyfluthrin 9% + imidacloprid 21% were given at 10 days intervals starting from the button stage at 0.025 and 0.05 per cent concentration. The initial deposits of 0.10 and 0.11 $\mu\text{g g}^{-1}$ for β -cyfluthrin and imidacloprid respectively, were recorded at standard dose of application. The corresponding levels at higher dose were 0.17 and 0.23 $\mu\text{g g}^{-1}$. The residues of β -cyfluthrin dissipated at relatively faster rate and

reached below determination limit on third day, whereas imidacloprid residues attained the below determination limit on seventh day at standard dose of application.

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⁻¹. Initial residues of imidacloprid on mango fruits from the two treatments were 0.329 and 0.536 mg kg⁻¹, respectively. Imidacloprid residues remained on mango fruits beyond 15 days and dissipated with the half-life of 5.2 and 8.2 days.

Reddy *et al.* (2013) studied residue dynamics of imidacloprid and hexaconazole on mango. Three sprays of imidacloprid 17.8 SL at 125 ml ha⁻¹ and 250 ml ha⁻¹ while hexaconazole 5 SL at 100 ml ha⁻¹ and 200 ml ha⁻¹ were given at panicle emergence, marble stage and maturity stage. Initial deposits of imidacloprid at single and double dose treated samples were 0.52 and 0.83 ppm respectively, which dissipated to Below Detectable Level (BDL) at 5 and 7 days after last spray. 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.

Phartial and Shrivastava (2014) studied dissipation of imidacloprid on lemon fruit. Imidacloprid 17.8 per cent SL was applied at the rate of 0.008 per cent on citrus fruits. Mean initial deposits was found 2.66 mg kg⁻¹ 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 upto 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⁻¹ and it was dissipated upto 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 per

cent 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 residues in brinjal. Residues of imidacloprid at 20 g a.i. ha⁻¹ and lambda cyhalothrin at 15 g a.i. ha⁻¹ were estimated in/on brinjal fruits. Initial deposits of in of imidacloprid dissipated to 93.17 per cent on 10th day. In lambda cyhalothrin the initial deposits were 0.138 mg kg⁻¹ which dissipated to 92.75 per cent on 10th day. The degradation of imidacloprid was relatively higher as compared to lambda cyhalothrin. 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 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⁻¹).

2.1.4 Thiomethaxam

Munj (2011) carried out an experiment to determine pre harvest interval of thiomethaxam in Alphonso mango fruits. Thiomethaxam 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 thiomethaxam residue at 12 days after spray. Thus, he suggested 12 days of pre harvest interval for thiomethaxam.

Tahany *et al.* (2011) studied residual behavior of thiomethaxam in kidney bean and tomato fruits. The initial deposits of thiomethaxam in kidney bean were 0.29 ppm which declined with the half-life of 4.14 days. The safe period of 7 days was recorded to consume fruits safely.

Chauhan *et al.* (2013) carried out a field experiment to study dissipation pattern of thiomethaxam on okra fruits. thiamethoxam was applied at recommended dose i.e. 25 g a.i. ha⁻¹ at fruiting stage of okra showed that the initial deposits of

0.245 mg kg⁻¹ reached below detectable level of 0.005 mg kg⁻¹ at 15 days after application with a half-life period of 1.47 days.

Alrahman (2014) carried out field experiments to study residue and dissipation kinetics of thiomethoxam in potato. Thiamethoxam (Actara 25% WG) was sprayed at the recommended dose (20 g 100 L⁻¹ water). The average residue of thiamethoxam was 2.16 mg kg⁻¹ 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⁻¹. 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⁻¹ after 5 days of its application at the recommended dosage.

2.2 Persistence of insecticides in fruits and vegetables

2.2.1 Cypermethrin

Awasthi (1985) reported longer persistence of synthetic pyrethroids on mango fruits, but based on tolerance limit as well as persistence pattern, the waiting periods of 3 to 6 days for cypermethrin, 6 to 9 days for permethrin and 11 to 12 days for fenvalerate could be followed, while deltamethrin treatments were found to leave residues within safe limit at the initial stage itself and as such did not need any waiting period. Pyrethroids being highly non-polar were found on the fruit pericarp only, therefore peeling off the fruit skin effectively dislodged the residues.

Awasthi (1986) studied persistence pattern and safety evaluation of synthetic pyrethroids on grapes and reported the waiting period of 2.6 days for permethrin, nil days for cypermethrin, 9.5 days for fenvalerate, nil days for deltamethrin, 5.3 days for dichlorovos and 13.5 days for monocrotophos with concentration of permethrin at 0.05 and 0.02 per cent, cypermethrin at 0.0075 and 0.01 per cent, fenvalerate at 0.015 and 0.02 per cent, deltamethrin at 0.0015 and 0.002 per cent, dichlorovos at 0.10 and 0.20 per cent and monocrotophos at 0.05 and 0.10 per cent.

Awasthi (1987) carried out field trial to study residues of synthetic pyrethroid on cabbage heads. Insecticides viz. permethrin, fenvalerate, cypermethrin and deltamethrin were applied at their effective application rates. Pyrethroid

residues were found dissipating at the half-life values of 2.6 to 2.9 days for permethrin, 2.7 to 2.8 days for fenvalerate, 1.9 to 2.5 days for cypermethrin and 1.6 to 3.0 days for deltamethrin respectively. The waiting periods of 11 to 13 days for permethrin, 11 to 14 days for fenvalerate, 6 to 9 days for cypermethrin and 2 to 4 days for deltamethrin.

Awasthi (1988) studied persistence of 4 synthetic pyrethroids residues 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.

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⁻¹. 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.

Jain *et al.* (2012) conducted an experiment to study the persistence and dissipation of cypermethrin, propanophos and ethion in/on soyabean. Cypermethrin was applied at the dose of 100 to 200 g a.i. ha⁻¹. Propanophos was applied at the dose of 1000 to 2000 g a.i. ha⁻¹ and ethion was applied at the dose of 1000 to 2000 g a.i. ha⁻¹. In all cases insecticides were persisted more than 11 days on leaves and 9 days on pods. The desirable waiting period were worked out to be 5.50 to 7.46 days (leaves) and 5.37 to 7.23 days (pods) for cypermethrin, 6.71 to 7.86 days (leaves) and 6.27 to 7.73 days (pods) for propanofos and 5.27 to 6.58days (leaves) and 4.37 to 5.96 days (pods) for ethion.

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⁻¹ and 2.84 and 4.54 mg kg⁻¹ 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 b) studied persistence pattern of chlorpyrifos, cypermethrin and monocrotophos on brinjal. The pesticides were applied at the dose of 100, 200 and 300 g a.i. ha⁻¹ on brinjal. Fruits were collected on 0,1,3,5,7,9,11,13,15 and 17 days after treatment of pesticides. The average initial residues of chlorpyrifos, cypermethrin and monocrotophos were in the range of 0.362-0.876, 0.340-0.858 and 0.388-0.891 mg kg⁻¹ respectively. The residues fell below detection in 13-17, 11-15 and 13-17 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⁻¹ at 10 days interval. The average initial deposits of cypermethrin in chilli fruits were found to be 1.46 and 3.11 mg kg⁻¹ 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⁻¹ after 25 days at both the application doses.

Banshtu and Patyal (2016) carried out study to compare the persistence of endosulfan and cypermethrin in ready mix formulation of Endohyper 40 EC (endosulfan 35%+ cypermethrin 5%) with individual insecticide endosulfan (Endocel 35 EC) and cypermethrin (Challenger 25 EC). Insecticides were applied at recommended dose of 350 and 500 g a.i. ha⁻¹ and at double recommended dose of 700 and 1000 g a.i. ha⁻¹ on cauliflower crop. In curds the endosulfan and

cypermethrin residues reached below detectable limit in 15 and 7 days when applied at recommended rate while when they applied at double recommended rate the residues reached below detectable limit in 20 and 10 days respectively. Endosulfan residues required 1.81-2.32 days and cypermethrin deposits required 1.19-1.44 days to reduce to half as a combination product, but when applied individually, half-life value for endosulfan is 1.21-1.51 days and cypermethrin deposits become half in 1.38-1.69 days.

2.2.2 Quinolphos

Aktar *et al.* (2008) studied degradation and persistence of quinolphos in/on okra fruits. Quinolphos was applied at 21 days after spraying by foliar spray at recommended and recommended dose (i.e. 500 and 1000 g a.i. ha⁻¹). The initial residues were found to be 3.20 and 7.50 µg g⁻¹ which dissipated with the half-life of 1.25 and 1.43 days at the dose of 500 and 1000 g a.i. ha⁻¹ respectively.

Aktar *et al.* (2010) studies risk assessment and decontamination of quinolphos in cabbage. Quinolphos 20 AF was applied at the rate of 500 and 1000 g a.i. ha⁻¹ in cabbage. The samples were harvested at an interval of 0 (3 hrs after application), 2, 4, 6, 8 and 10 days interval. The calculated half-life values were 1.27 and 1.38 for cabbage heads. Whereas, safe waiting period was 5.28 and 6.7 days.

2.2.3 Imidacloprid

Gajbhiye *et al.* (2000) studied translocation and persistence of imidacloprid in tomato following seed and root dip treatments. Imidacloprid was found to translocate from seed and root to the leaves. The translocated residues persisted for 45 days after transplanting. However, no residues were detected in tomato fruits.

Gupta *et al.* (2005) conducted an experiment to study persistence of imidacloprid in gram. Imidacloprid was applied at the rate of 20 and 40 g a.i. ha⁻¹ by foliar spray. The residues of imidacloprid persisted beyond 3 days but no residues were detected on 5th day.

Mahopatra *et al.* (2011) studied behaviour of beta cyfluthrin and imidacloprid in mango. Insecticides were applied as a mix formulation, beta cyfluthrin 9%+ imidacloprid 21% (Solomon 300 OD) at the concentration of 75 and 150 g a.i. ha⁻¹. Initial deposits of beta cyfluthrin dissipated with the half-life of 2.4 and 2.6 days at the concentration of 75 and 150 g a.i.ha⁻¹ respectively and persisted for 5 days only. Imidacloprid residue degraded with the half-life of 3.06 and 4.16 days at the concentration of 75 and 150 g a.i. ha⁻¹ respectively and persisted for 10 days.

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⁻¹ in the fruits of brinjal, tomato and okra. The study indicated 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 is lower than that of imidacloprid in fruits of these vegetables. Moreover, the persistence of these insecticides when compared between different fruits, it is 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⁻¹. 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⁻¹) and higher dose (54 g a.i. ha⁻¹) respectively.

Bhattachergy (2013) studied persistence behaviour of imidacloprid and carbosulfan in mango. Imidacloprid was sprayed at the concentration of 0.3 ml L⁻¹ of water and carbosulfan was sprayed at 2.0 ml L⁻¹ of water. Residues of both insecticides were analysed in peel, pulp and fruit at different stages of fruit development and maturity. The initial residues of imidacloprid after 30 days of spraying, persisted for 60 and 50 days in peel and pulp respectively and dissipated with the half-life of 38 days. Mature fruits at harvest (after 85 days of spraying) were free from imidacloprid residues. Carbosulfan in mango peel dissipated from

5.30 to 0.05 mg kg⁻¹ at the time of harvest. The insecticides residue was not detected in pulp at the time of harvest. The residue persisted for 26 and 45 days in pulp and peel respectively and degraded with the half-life of 7 days. Therefore, the safe pre harvest intervals were suggested to be 55 and 46 days for imidacloprid and carbosulfan respectively.

2.2.4 Thiomethaxam

Gupta *et al.* (2005) conducted an experiment to study persistence of thiomethaxam in gram. Thiomethaxam was applied at the concentration of 3 and 6 g a.i. kg⁻¹ of seed. The residues of thiomethaxam persisted beyond 5 days but no residues were detected on 10th day except for high dose of thiomethaxam.

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⁻¹ and the double rate of 280 g a.i. ha⁻¹ was carried out 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.

Bhattachargee and Dikshit (2016) studied dissipation kinetics and risk assessment of thiomethaxam and dimethoate in mango. Thiamethoxam (0.008 and 0.016 %) and dimethoate (0.06 and 0.12 %) were sprayed on mango trees during pre-mature stage of fruits. Thiamethoxam dissipated in fruit from 1.93 and 3.71 mg kg⁻¹ after 2 hours of spraying to 0.08 and 0.13 mg kg⁻¹ 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⁻¹ after 2 hours of application to 0.12 and 0.19 mg kg⁻¹ after 10 days of application at single and double doses respectively. No residue was detected in fruit beyond 10 days after its application. In both cases mature fruits (after 40 days of spraying) were free from any residue of these insecticides at both the concentration levels. The pre harvest intervals of 7 to 11 days for thiomethaxam and 6 to 7 days for dimethoate were recorded.

Hafez and Singh (2016) studied persistence of thiomethaxam in/on tomato fruits and soil. Dissipation behaviour of thiamethoxam in tomato fruits and soil was studied following application with recommended dose (50 g a.i. ha⁻¹) and double recommended dose (100 g a.i. ha⁻¹). The initial deposits in tomato fruits (1 h after spraying) were found to be 0.11 µg g⁻¹ and 0.18 µg g⁻¹ at recommended and double recommended dose, respectively. While the initial deposits in soil were found to be 0.06 µg g⁻¹ and 0.11 µg g⁻¹ at recommended dose and double recommended dose, respectively. Residues of thiamethoxam reached below determination limit at 7th day and 10th day in tomato fruits and 7th 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.

2.3 Nutrient content in Mango fruits (N, P, K, Ca, Mg, S, Fe, Mn, Zn, Cu).

2.3.1 Total macronutrient content in fruits of Mango orchards:

Dhopavkar (2001) found that the N, P and K contents in fruits were 0.637, 0.079 and 0.389 per cent respectively.

Patil *et al.* (2010) analysed the Nitrogen, Phosphorous and potassium percentage ranging from 0.992-1.10, 0.01-0.03 and 0.37-0.55, respectively.

Dabke *et al.* (2013) reported that the N, P and K contents in fruit pulp were 0.96, 0.068 and 0.56 per cent, respectively.

Joshi (2015) reported that the macronutrient content in Alphonso mango fruits were 0.223 per cent (N), 0.03 per cent (P), 0.435 per cent (K).

Puranik (2015) from his study on 'Periodical nutrient content in soil and leaf of Alphonso mango orchards from Ratnagiri and Devgad and their effect on yield and quality' showed the values of total N, P, K, Ca, Mg and S content as 0.97 per cent, 0.10 per cent, 0.56 per cent, 0.44 per cent and 0.44 per cent and 0.40 per cent respectively.

Thakare (2016) from her study on 'Effect of long term fertilizer management practices on yield and quality of mango'showed the values of total N, P, K, Ca, Mg, and S as 0.55 per cent, 0.13 per cent, 0.75 per cent, 0.45 per cent, 0.20 per cent and 0.58 per cent respectively.

2.3.2 Total micronutrient content in fruits of Mango orchards:

Patil *et al.* (2010) analysed the Fe, Mn, Zn and Cu content of mango fruits ranging from, 88.13-161.33, 2.90-9.93 ppm, 36.76-79.96, and 4.13-12.73 ppm, respectively.

Joshi (2015) reported that the micronutrient content in Alphonso mango fruits were 30.62 $\mu\text{g g}^{-1}$ (Fe), 22.67 $\mu\text{g g}^{-1}$ (Mn), 9.16 $\mu\text{g g}^{-1}$ (Zn) and 14.10 $\mu\text{g g}^{-1}$.

Puranik (2015) from his study on 'Periodical nutrient content in soil and leaf of alphonso mango orchards from ratnagiri and devgad and their effect on yield and quality' showed the values of total Fe, Mn, K, Zn and Cu content as 30.62 $\mu\text{g g}^{-1}$, 22.67 $\mu\text{g g}^{-1}$, 9.16 $\mu\text{g g}^{-1}$ and 14.10 $\mu\text{g g}^{-1}$ respectively.

Thakare (2016) from her study on 'Effect of long term fertilizer management practices on yield and quality of mango'showed the values of total Fe, Mn, Zn and Cu as 121.37 $\mu\text{g g}^{-1}$, 87.72 $\mu\text{g g}^{-1}$, 16.85 $\mu\text{g g}^{-1}$ and 13.63 $\mu\text{g g}^{-1}$ respectively.

CHAPTER III

MATERIAL AND METHODS

The present investigation, entitled, “Estimation of insecticidal residue in Alphonso mango fruits” was conducted at mango orchard on the farm of department of Agronomy, College of Agriculture, Dapoli and the mango orchard belongs to the private farmer in the year 2015-16. The analytical work was done in the research laboratory of the Department of Soil Science and Agricultural Chemistry, College of Agriculture, Dapoli, College of Forestry, Dapoli, Dist. Ratnagiri and Chemical research laboratory, Ross lifescience Pvt. Ltd. Pune (M.S.). The details regarding material and methods used for analytical works are presented in this chapter.

3.1 Material

3.1.1 Geography and Agro-climatic conditions:

The domain of the present research work comes under the Ratnagiri district located in south western part of Maharashtra state on the Arabian Sea coast. The district is identified as Very High Rainfall Lateritic (VRL) zone of agro-climatic regions of Maharashtra. Dapoli is located at 17.45° N latitude, 73.10° E longitude and 250 m above mean sea level. The climate is hot, humid with well-expressed three seasons *viz.* summer (March to May), rainy (June to October) and winter (November to February). The mean annual rainfall at Dapoli is 3500 mm, which is generally received from June to October with about 95 to 100 rainy days. The weather parameters recorded at the Meteorological Observatory, College of Agriculture, Dapoli during present study are mentioned in Table 3.1

3.1.2 Experimental site

a) Orchard 1: Department of Agronomy, College of Agriculture, Dapoli.

Dist. Ratnagiri (M.S.)

b) Orchard 2: Mr. Shrikrushna Phatak, Jalgaon (Dapoli), Dist. Ratnagiri

(M.S.)

Table 3.1: The weather parameters recorded during *Summer* season of 2016 at Meteorological Observatory, Department of Agronomy, College of Agriculture, Dapoli

Period (2016)	Temp (°C)		Relative Humidity (%)		Sun Shine (hrs. day ⁻¹)	Rainy Day
	Max.	Min.	Morning	Evening		
12.02 - 18.02	29.2	14.0	93	50	8.5	0
19.02 - 25.02	32.8	16.8	90	55	8.2	0
26.02 - 04.03	33.9	18.7	92	61	6.9	0
05.03 - 11.03	34.4	16.1	89	47	8.2	0
12.03 - 18.03	31.4	16.4	92	55	8.7	0
19.03 - 25.03	35.7	18.4	87	62	7.9	0
26.03 - 01.04	33.7	18.7	92	61	8.2	0
02.04 - 08.04	32.9	20.0	85	69	7.1	0
09.04 - 15.04	34.8	20.1	84	75	8.9	0
16.04 - 22.04	32.6	21.3	91	65	8.8	0
23.04 - 29.04	33.4	20.3	91	66	9.7	0
30.04 - 06.05	33.9	21.0	91	65	10.0	0
07.05 - 13.05	34.3	23.5	90	67	9.2	0
14.05 - 20.05	33.5	24.8	88	69	7.2	0
21.05 - 27.05	34.2	25.9	84	64	9.0	1
28.05 - 03.06	34.7	25.6	83	59	9.2	1

3.1.3 Insecticides

Table 3.2 Insecticides used in mango orchard 2

Sr.No.	Name of insecticide	Chemical Name
1	Cypermethrin 25 EC	RS-cyano-3 phenoxy benzyl (1RS)-cis-trans-(2,dichlorovinyl)2,2-dimethylcyclopropane carboxylate
2	Thiomethaxam 25 EC	(NE)-N-[3-[(2-chloro-1,3-thiazoyl)methyl]-5-methyl-1,3,5-oxadiazinan-4-ylidene]nitramide

Table 3.3 Insecticides used in mango orchard 1

Sr. No.	Name of Insecticide	Chemical Name
1	Cypermethrin 25EC	RS-cyano-3 phenoxy benzyl (1RS)-cis-trans-(2,dichlorovinyl)2,2-dimethylcyclopropane carboxylate
2	Quinolphos 25 EC	0,0-dimethyl-S-2-(ethyl-thio)ethyl phosphonothioate
3	Imidacloprid 17.8 EC	1-(6-chloro-3-pyridylmethyl)-N-nitro maidazolidin-2-ylideamine
4	Thiomethaxam 25EC	(NE)-N-[3-[(2-chloro-1,3-thiazoyl)methyl]-5-methyl-1,3,5-oxadiazinan-4-ylidene]nitramide

3.1.4Appliance

Manually operated foot sprayer was used for spraying the mango trees.

3.1.5 Field experiment

3.1.5.1 Experiment 1

In this experiment thirty mango trees were randomly selected from the mango orchard 1. The spraying was carried out as per the recommended schedule of insecticides on all thirty trees. Mango fruit samples were collected randomly from each tree at egg stage as well as at harvest stage. The details regarding insecticidal spray schedule and dates of collection of fruit sample are presented in

3.1.5.2 Experiment 2

In this experiment thirty mango trees were selected from the mango orchard 2. The spraying was carried out as per their own schedule on all thirty trees. Mango fruit samples were collected randomly from each tree at harvest stage. The details regarding insecticidal spray schedule and dates of collection of fruit samples are presented in **Table 3.5**

Table 3.4 Insecticidal spray schedule followed on orchard 1 and dates of collection of mango fruit samples at egg stage and at harvest stage.

Spray	Name of insecticides	Time of spray	Quantity per 10 lit of water	Date of application	Date of sampling	
					Egg Stage	Harvest stage
1 st	Cypermethrin 25 EC	At vegetative flush stage	3 ml	30/10/015	19/03/016	27/05/016
2 nd	Quinolphos 25 EC	At flowering stage	20 ml	06/12/015	19/03/016	27/05/016
3 rd	Imidacloprid 17.8 EC	Just before fruit setting stage	3 ml	05/01/016	19/03/016	27/05/016
4 th	Thiomethaxam 25 EC	15 days after third spray	1 gm	03/03/016	19/03/016	27/05/016

Table 3.5 Insecticidal spray schedule followed on orchard 2 and dates of collection of mango fruit samples at harvest stage.

Spray	Name of insecticides	Time of spray	Quantity per 10 lit of water	Date of application	Date of sampling at harvest
1 st	Cypermethrin 25 EC	At vegetative flush stage	10 ml	27/10/2015	25/5/016
2 nd	Thiomethaxam 25 EC	At fruit setting stage	4 gm	26/ 2/ 2016	25/ 5 /016

3.2 Methods

3.2.1 Methods of residue analysis

Extraction method

Samples of fruits were randomly harvested from experimental trees. The samples were subjected to extraction on the same day of harvesting. The 50 g of finely chopped representative samples were macerated with 40 g unhydrous sodium sulphate and 15 g of sodium hydrogen carbonate in blending machine. After blending, the fine paste of mango samples were extracted with 200 ml of ethyl acetate by using mechanical shaker for 1 hr. The total content was filtered and concentrated upto 50 ml by using rotary evaporator (Alyaseri *et al.*,2012).

3.2.1.1 Estimation of Neonicotinoid insecticides (Thiomethaxam and Imidacloprid).

Ethyl acetate extracted samples were provided for analysis. One mL of extract in glass tube was evaporated on N₂ evaporator to dryness. Reconstituted the residue with 1 mL of acetonitrile, vortexed for 1 min and injected to UPLC-MSMS.

Residues of Thiomethaxam and Imidacloprid were estimated by UPLC-MSMS. Other details of UPLC-MSMS system are given below.

Instrumental details for analysis of thiomethaxam and imidacloprid

Instrument Name	:	UPLC-MS/MS
Instrument Model	:	XEVO TQD coupled with Acquity UPLC H class
Column	:	Acquity UPLC BEH C18, 1.7 μ m, 2.1*100 mm
Detector	:	MS/MS
Instrument Make	:	Waters
Mobile Phase-A	:	Water 0.1% formic acid
Mobile Phase-C	:	Methanol
Flow rate	:	0.3 mL/min
Run time	:	5 min

MS/MS Condition

Ionization mode	:	ESI Positive
Capillary Voltage (Kv)	:	3.3
Source Temperature (°C)	:	150
Desolvation gas Flow(L/Hr)	:	650

Retention time

Thiamethoxam	:	3.05 min
Imidacloprid	:	3.19 min

Note: The retention time of this insecticide was determined by injecting technical grade insecticide before the samples were injected.

3.2.1.2 Estimation of Cypermethrin

One ml of ethyl acetate extracted sample injected to GC–MSD for estimation of cypermethrin. other details of GC-MSD system are given below.

Instrumental details for analysis of Cypermethrin.

Instrument Name	:	GC-MSD
Instrument Serial No.	:	GC-CN13432012, MSD-US1344R205
Instrument Name	:	GC-MSD
Column	:	DB-5MS 30mX320µmX0.25µm
Detector	:	MASS DETECTOR
Instrument Make	:	Agilent
Column Flow	:	2 mL/min
Carrier Gas	:	Helium (Purity99.9%)

Working temperatures

Inlet temperature (°C)	:	250
Valve Temperature(°C)	:	200
Source Temperature (°C)	:	230
MSQuad Temperature (°C)	:	150

Retention time : 23.3 min.

Note: The retention time of this insecticide was determined by injecting technical grade insecticide before the samples were injected.

3.2.1.3 Estimation of Quinolphos

One ml of ethyl acetate extracted sample injected to GC–MSD for estimation of Quinolphos. other details of GC-MSD system are given below.

Instrumental details for analysis of Quinolphos

Instrument Name	:	GC-MSD
Instrument Serial No	:	GC-CN13432012, MSD-US1344R205
Instrument Name	:	GC-MSD
Column	:	DB-5MS 30mX320µmX0.25µm
Detector	:	MASS DETECTOR
Instrument Make	:	Agilent
Column Flow	:	2 mL/min
Carrier Gas	:	Helium (Purity99.9%)

Working temperatures

Inlet temperature (°C)	:	250
Valve Temperature(°C)	:	200
Source Temperature (°C)	:	230
MSQuad Temperature (°C)	:	150

Retention Time	:	3.9 min.
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Note: The retention time of this insecticide was determined by injecting technical grade insecticide before the samples were injected.

3.2.2 Methods of fruit analysis

Table 3.5 Methods of fruit nutrient analysis.

Sr.No	Properties	Name of Method	Described by
1	Total Nitrogen	Micro-Kjeldahl method	Tandon (1993)
2	Total Phosphorous	Vanado –molybdate yellow colour method	Tandon (1993)
3	Total potassium	Flame photometry method	Tandon (1993)
4	Calcium and Magnesium	Versanate titration	Chopra and Kanwar, 1978
5	Total Sulphur	Turbidimetric method	Cheshin and Yien, (1950)
6	Micronutrient Fe, Mn, Zn, Cu	Atomic Absorption Spectrophotometry method	Mclaren and Crawford (1950)

3.3 Method of calculation of insecticidal residue.

3.3.1 Residues in ppm or mg/kg

The parts per million (ppm) or mg/kg of cypermethrin, quinolphos, imidacloprid and thiomethaxam residues in mango fruits were calculated by following formula.

$$\text{Residues (ppm)} = \frac{F_A \times V_{\text{end}} \times W_{\text{st}}}{F_{\text{st}} \times V_i \times G}$$

Where,

G = Weight of analytical sample (g)

V_{end} = Final volume of the sample solution (ml)

V_i = Aliquot volume of V_{end} injected in GC (μl)

F_A = Peak area/height of the analytical sample solution obtained from V_i

F_{st} = Peak area/height of the standard solution obtained from W_{st}

W_{st} = Amount of the reference standard substance injected with the standard solution in (μg)

Statistical analysis

The data have been subjected to appropriate method of statistical analysis as described by Panse and Sukhatme (1967).

CHAPTER IV

RESULTS AND DISCUSSION

The present investigation encompasses variety of data related to insecticidal residue in Alphonso mango fruits from the orchard located at Department of Agronomy, College of Agriculture, Dapoli and Mango orchard belonging to private farmer. The data presented attempts to associate the insecticidal residue content of mango fruits at these two locations. In addition, the study endeavors persistence of insecticides. The results obtained are presented in appropriate tables, figures along with chromatograms and discussed under suitable headings.

4.1 Persistence of insecticides in mango fruits from orchard 1

4.2 Insecticidal residue in mango fruit samples at harvest from orchard 1

4.3 Insecticidal residue in mango fruit samples at harvest from orchard 2

4.4 Comparison between Insecticidal residue in mango fruits from orchard 1 and 2

4.5 Total nutrient content of mango fruit samples from orchard 1 and 2

4.1 Persistence of insecticidal residue in mango fruits from orchard 1

Table 4.1 Insecticide residues in Alphonso mango fruits at egg stage and at harvest stage, after application of insecticides individually as per the recommended schedule

Sr. No.	Insecticide	Conc.(%)	Egg Stage		Harvest Stage		MRL
			Sampling period after spay (days)	*Mean residue (ppm)	Sampling period after spay (days)	*Mean residue (ppm)	
1	Cypermethrin	0.0075	140	BDL (0.06)	208	BDL (0.05)	0.2
2	Quinolphos	0.05	103	BDL	172	BDL	0.2

				(0.07)		(0.05)	
3	Imidacloprid	0.0053	73	BDL (0.01)	143	ND	1
4	Thiomethaxam	0.0025	15	ND	84	ND	1

* Average of fruit samples; BDL – Below Detectable Limit; ND- Not Detected

The results of residue analysis of cypermethrin, quinolphos, imidacloprid and thiomethaxam in mango fruit samples from orchard 1 at egg stage and at harvest stage are given in **Table 4.1**.

4.1.1 Persistence of Cypermethrin

The persistence of cypermethrin was tested at the spray concentration of 0.0075%. Its residue in mango fruit samples at egg stage was 0.06 ppm that reduced to 0.05 ppm at harvest stage. (**Table 4.1**). Thus, it is evident from the data that cypermethrin residues in mango fruit samples at egg stage as well as at harvest stage were found below detectable level of 0.2 ppm.

The first spray of cypermethrin was given 140 days before egg stage. As reported by Awasthi (1985) the persistence of cypermethrin in mango fruits is only for 6 days from spraying. Similarly, Awasthi (1988) reported that cypermethrin 0.01 per cent persisted only for three weeks i.e. 21 days after spraying and showed that cypermethrin did not penetrate into the pulp at any stage of fruit. Moreover, Munj (2011) suggested 42 days of pre harvest interval for cypermethrin at 0.0075% concentration. Hence, residues of cypermethrin at 0.0075% concentration cannot persist in mango fruits for such a long period after spraying i.e. 140 and 208 days for egg stage and harvest stage respectively. Chromatograms of cypermethrin residue in mango fruit samples at egg stage and at harvest stage are presented in **Fig. 4.1**

4.1.2 Persistence of Quinolphos

The persistence of quinolphos was tested at the spray concentration of 0.05 per cent. Its residue in mango fruit samples at egg stage was 0.07 ppm which decreased upto 0.05 ppm at harvest stage. (**Table 4.1**). Thus, it is evident from the data that quinolphos residues were found below detectable level of 0.2 ppm at both the stages.

The egg stage fruits were sampled after 103 days of spraying of quinolphos (0.05%). The safe waiting periods of quinolphos for mango fruits are 11.5 and 14.2 days at 0.05 and 0.1 per cent dose respectively Vijayalakshmi (2002). While pre harvest interval of 42 days at 0.05 per cent concentration was reported by Munj (2011). Therefore, the residues of quinolphos were not expected to persist in mango fruits at egg stage (103 days after spraying) and at harvest stage (172 days after spraying). Chromatograms of quinolphos residues in mango fruit samples at egg stage and at harvest stage are presented in **Fig. 4.2**

4.1.3 Persistence of Imidacloprid

The persistence of imidacloprid was tested at the spray concentration of 0.0053 per cent. Its residue in mango fruit samples at egg stage was 0.01ppm which is below detectable level of 1 ppm which reduced to non detectable level at harvest stage of mango fruits (**Table 4.1**).

The egg stage fruits were collected after 73 days of spraying of imidacloprid at 0.0053% concentration. Earlier, Sharma and Awasthi (1998) reported that imidacloprid residue did not found in mango fruit pulp at harvest even at higher concentration. Mahopatra *et al.* (2011) reported that the residues of imidacloprid in mango fruits persisted for 10 days after spraying while, Bhattacharjee (2013) showed that the residues of imidacloprid persisted in mango fruits for 60 and 50 days in peel and pulp respectively. During present investigation mango fruits were collected after a long period after spraying of insecticides at fruit sample collection i.e. 73 days for egg stage and 143 days for harvest stage. Hence, the persistence of imidacloprid at 0.0053% concentration was not expected in mango fruits for such

a long interval. Chromatograms of imidacloprid residues in mango fruit sample at egg stage are presented in **Fig.4.3**

4.1.4 Persistence of Thiomethaxam

The persistence of thiomethaxam was tested at the spray concentration of 0.0025 per cent. Its residue in mango fruit samples was not detected at egg stage as well as at harvest stage (**Table 4.1**).

During present study, the fruit samples were collected at 15 days (egg stage) and 84 days (harvest stage) after spraying. While, Munj (2011) reported that mango fruit samples after spraying of thiomethaxam at 0.0025% concentration were found free from thiomethaxam residues at 12 days.

Thus, the residual persistence of thiomethaxam was not expected in mango fruits after such a long interval. Chromatogram of thiomethaxam standard is given in **Fig. 4.4**

4.2 Insecticidal residue in mango fruit samples at harvest from orchard 1

Table 4.2 Insecticide residues in Alphonso mango fruits at harvest stage after application of recommended schedule of insecticides (Orchard 1)

Sr. No.	Insecticide	Sampling period after spray (days)	*Mean residue (ppm)	MRL (ppm)
1	Cypermethrin (0.0075%)	208	BDL (0.05)	0.2
2	Quinolphos (0.05%)	172	BDL (0.05)	0.2
3	Imidacloprid (0.005%)	143	ND	1
4	Thiomethaxam (0.0025%)	84	ND	1

4.2.1 Monitoring of cypermethrin residues in mango fruits

Cypermethrin 25 EC was evaluated for its residue in harvest stage mango fruits. It was tested at the spray concentration of 0.0075 per cent as a recommended dose. It is evident from the data in **Table 4.2** that the residual value of cypermethrin at 0.0075 per cent concentration in mango fruit samples was 0.05 ppm which was below detectable level of 0.2 ppm.

The spraying of cypermethrin was undertaken during the month of October and harvest stage sampling was done during the month of May (**Table 3.4**) So, there is longer period of gap between spraying and harvest time sampling i.e. 208 days (**Table 4.2**). Earlier, Munj (2011) and Dubale *et al.* (2011) found that spraying of cypermethrin at 0.0075 per cent concentration did not show any detectable residues in harvest stage fruits. Furthermore, Awasthi (1985 and 1988) showed that cypermethrin found on fruit pericarp only and it did not penetrate into the pulp of fruit at any stage after spraying. Residues of cypermethrin in okra reached below detection in 17 days (Chandra *et al.*, 2014).

Moreover, during fruit development process there might be faster degradation of the residues and that too in summer because of high temperature i.e. 34.12°C average maximum temperature in the month of May (**Table 3.1**). This might have lead to complete breakdown of cypermethrin residues. Their faster degradation during fruit development period probably did not allow its residual persistence in fruit at harvest. Chromatograms for cypermethrin standard as well as for cypermethrin residue in egg stage and in harvest stage fruit samples are presented in **Fig.4.1**

4.2.2 Monitoring of Quinalphos residues in mango fruits

Quinalphos 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the spray concentration of 0.05 per cent as a recommended dose. It is evident from the data in **Table 4.2** that mean residual value of

quinolphos at 0.05 per cent was 0.05 ppm which was below detectable level of 0.2 ppm.

The organophosphate molecule, quinolphos (0.05%) was applied on mango trees during the month of December and mango fruit samples were collected in the month of May (**Table 3.4**). So, there is 172 days of gap between the application of quinolphos and harvest time sampling of mango fruits (**Table 4.2**). While, Vijayalakshi (2002) recorded 11.5 and 14.2 days of waiting period and Munj (2011) suggested 42 days of pre harvest interval for safe consumption of mango fruits. Similar result have been recorded by Dubale *et al.*, (2011) with the application of quinolphos at 0.05 per cent concentration. As organophosphate insecticides do not persist in plants for longer time, the residues of quinolphos did not persist in mango fruits for such a long interval. Chromatograms for quinolphos standard as well as for quinolphos residues in egg stage and in harvest stage fruits are given in **Fig. 4.2**

4.2.3 Monitoring of Imidacloprid residues in mango fruits

Imidacloprid 17.8 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the spray concentration of 0.0053 per cent as a recommended dose. The data given in **Table 4.2** showed that spraying of imidacloprid at 0.0053 per cent concentration did not show any detectable residue in mango fruit samples at harvest stage. Dubale *et al.* (2011) reported that the residues of imidacloprid at 0.0053 per cent concentration in mango fruits were below detectable level at harvest. Similarly, Sharma and Awasthi (1998) revealed that at harvest, residues of imidacloprid in mango fruits were below detectable level even at higher concentration.

Imidacloprid (0.005%) was applied during the month of January and mango fruit samples were collected during the month of May (**Table 3.4**). So, there is 143 days of gap between the spraying and at harvest (**Table 4.2**). Moreover, during

fruit development process there might be faster degradation of the residues and that too in summer i.e. 34.12°C average maximum temperature in the month of May (**Table 3.1**). This might have lead to complete breakdown of imidacloprid residue in mango fruits at harvest. Their faster degradation during fruit development period probably did not allow its residual persistence in fruit at harvest. Chromatograms for imidacloprid standard and for its residue in egg stage fruits are presented in **Table 4.3**

4.2.4 Monitoring of Thiomethaxam residues in mango fruits

Thiomethaxam 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the spray concentration of 0.0025 per cent as a recommended dose. Spraying of thiomethaxam at 0.0025 per cent concentration did not show any detectable residue in mango fruit samples at harvest stage (**Table 4.2**).

Thiomethaxam (0.0025%) was applied during the month of March and the mango fruit samples were collected in the month of May (Table 3.4). So, there is 84 days of gap between the spraying and collection of fruit samples at harvest stage (**Table 4.2**). Earlier, Munj (2011) suggested that mango fruit samples were free from thiomethaxam residues after 12 days of spraying at 0.0025% concentration. While, Tahany *et al.*(2011) recorded 7 days of waiting period for safe consumption of kidney bean and tomoto fruits. So presence of thiomethaxam residue was not expected after such a long interval. Chromatogram of thiomethaxam standard is presented in **Fig. 4.4**

These results indicated that the above insecticides viz. cypermethrin (0.0075%), quinolphos (0.05%), imidacloprid (0.0053%) and thiomethaxam (0.0025%) applied individually as per the recommended schedule can be considered as a safe from insecticide residue point of view at harvest stage of fruits

4.3 Insecticidal residue in mango fruit samples at harvest from orchard 2

Table 4.3 Insecticide residues in Alphonso mango fruits at harvest stage after application of insecticides (Orchard 2)

Sr.No	Insecticide	Sampling period after spray (days)	*Mean residue (ppm)	MRL (ppm)
1	Cypermethrin (0.025%)	210	BDL (0.05)	0.2
2	Thiomethaxam (0.01%)	112	ND	1

4.3.1 Monitoring of cypermethrin residues in mango fruits

Cypermethrin 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the concentration of 0.025 per cent. The data presented in **Table 4.3** showed that, the mean residual value of cypermethrin at the spray concentration of 0.025 per cent was 0.05 ppm which was below detectable level of 0.2 ppm. While 3 to 6 days of waiting period for mango fruits suggested by Awasthi (1985). Moreover, Dubale *et al.* (2010) reported that 2 to 3 days of waiting period are required for safe consumption of mango fruits. Awasthi (1986) suggested '0' day (nil) waiting period for grapes when fruits were treated with 0.0075 per cent cypermethrin.

During present investigation cypermethrin was sprayed at the concentration of 1 ml L⁻¹ which is much more as compared to orchard 1 where cypermethrin was sprayed at the rate of 0.3 ml L⁻¹ (**Table 4.4**) while mango fruits were sampled after 210 days of spraying. As far as the above results are taken into account, it was not expected to found any residue of cypermethrin at such a long interval even at higher concentration.

4.3.2 Monitoring of thiomethaxam residues in mango fruits

Thiomethaxam was evaluated for residues in harvest stage of mango fruits. It was tested at the concentration of 0.01 per cent. The data presented in **Table 4.3** showed that, spraying of thiomethaxam at 0.01 per cent concentration did not show any detectable residues at harvest. Earlier, Munj (2011) suggested that mango fruit samples were free from thiomethaxam residues after 12 days of spraying at 0.0025 per cent concentration. Chauhan *et al.* (2013) showed that initial deposits of thiomethaxam reached below detectable level at 15 days after application on okra fruits.

During present investigation thiomethaxam was sprayed at the concentration of 0.4 gm L^{-1} which is much more as compared to orchard 1 where thiomethaxam was sprayed at the rate of 0.1 gm L^{-1} (**Table 4.4**) however mango fruits were sampled after 112 days of spraying. As far as the above results are taken into account, it was not expected to found any residue of thiomethaxam at such a long interval even at higher concentration.

4.4 Comparison between insecticidal residue from orchard 1 and 2

Table 4.4 Comparison between insecticidal residue level in mango fruits at harvest from both orchards

Sr.No.	Insecticide	Dose		* Mean residue(ppm)		MRL
		Orchard 1	Orchard 2	Orchard 1	Orchard 2	
1	Cypermethrin	0.3 ml L^{-1}	1 ml L^{-1}	BDL (0.05)	BDL (0.05)	0.2
2	Thiomethaxam	0.1 g L^{-1}	0.4 g L^{-1}	ND	ND	1

The comparison between insecticidal residue levels from the orchard 1, where insecticidal application schedule given by Dr. B.S.K.K.V. Dapoli was followed and the orchard 2 where private farmer followed their own schedule is given in **Table 4.4**.

It is evident from **Table 4.4** that cypermethrin and thiomethaxam were used in higher concentration in orchard 2 as compared to orchard 1. In spite of having higher dose, cypermethrin residues were found below detectable limit of 0.2 ppm in both the cases. Similarly, thiomethaxam residues were not detected in any fruit samples from both mango orchards. It may be due to long interval between spraying of insecticides and collection of fruit samples (**Table 4.2 and 4.3**).

The results thus indicated that the insecticide spray schedule, recommended for management of pests infesting mango blossom by Dr. B.S.K.K.V., Dapoli as well as insecticide spray schedule followed by private farmer are safe from pesticide residue point of view.

4.5 Primary, secondary and micronutrient content in mango fruits samples from orchard 1 and 2 at harvest stage.

Table 4.5 Macronutrient content of mango fruits at harvest from orchard 1 and 2

Sr.No.	Parameter	Orchard 1	Orchard.2
1	Total N (%)	0.55 (0.06-1.18)	0.49 (0.03-1.10)
2	Total P (%)	0.19 (0.11-0.29)	0.17 (0.14-0.23)
3	Total K (%)	0.52 (0.42-0.65)	0.60 (0.42-0.77)
4	Calcium (%)	0.33 (0.24-0.52)	0.28 (0.14-0.39)
5	Magnesium (%)	0.12 (0.04-0.28)	0.10 (0.02-0.26)
6	Total S (%)	0.43 (0.21-0.61)	0.37 (0.2-0.47)

As plant protection measures are important to obtain better yield of mango, yield of mango trees from both orchard were recorded. The average yield of mango fruits from orchard 1 was 7 tonnes ha⁻¹ and from orchard 2 was 9 tonnes ha⁻¹. As far as plant nutrition is concerned, primary, secondary and micronutrient content of mango fruit samples at harvest stage from both orchards were recorded and are given in **Table 4.5** and **4.6**.

4.5.1 Primary and secondary nutrient content in mango fruits at harvest.

The data related to primary and secondary nutrient content in mango fruits at harvest from both the orchards are presented in **Table 4.5** and depicted in **Fig.4.5**

Primary nutrient (Total N, P and K) content in mango fruits

The total nitrogen, phosphorus and potassium of harvest stage fruits as 0.06 to 1.18 with a mean of 0.55 per cent N, 0.11 to 0.29 with a mean value of 0.19 per cent P and 0.42 to 0.65 with a mean value of 0.52 per cent K in orchard 1

In orchard 2, the total nitrogen, phosphorus and potassium of harvest stage fruits were 0.03 to 1.10 with a mean value of 0.49 per cent N, 0.14 to 0.23 with a mean value of 0.17 per cent P and 0.42 to 0.77 with a mean value of 0.60 per cent K.

Somewhat similar values of N, P and K contents were reported by Dhopavkar (2001), Patil *et al.* (2010), Dabke *et al.* (2013), Joshi (2015), Puranik (2015) and Thakare (2016) in Alphonso mango fruits.

Secondary nutrients (Total Ca, Mg and S) content in mango fruits.

The data presented in **Table 4.5** when studied revealed that the total calcium, magnesium and sulphur content in mango fruits from orchard 1 ranged from 0.24 to 0.52 with the mean of 0.33 per cent Ca, 0.04 to 0.28 with a mean value of 0.12 per cent Mg and 0.21 to 0.61 with a mean value of 0.43 per cent S.

As seen from **Table 4.5** the total Calcium, Magnesium and Sulphur content of mango fruits from orchard 2 ranged from 0.14 to 0.39 with a mean value of 0.28 per cent Ca, 0.02 to 0.26 with a mean value of 0.10 per cent Mg and 0.2 to 0.47 with a mean value of 0.37 per cent

Similar values of Ca, Mg and S contents were reported by Puranik (2015) and Thakare (2016) Alphonso mango fruits in lateritic soils of Konkan.

4.5.2 Micronutrient content in mango fruits at harvest.

Table 4.6 Micronutrient status of mango fruits at harvest from orchard 1 and 2

Sr.No.	Parameter	Orchard 1	Orchard 2
1	Total Fe ($\mu\text{g g}^{-1}$)	42.3 (10.8-77.7)	38.3 (17.5-73.7)
2	Total Mn ($\mu\text{g g}^{-1}$)	16.3 (10.5-29.2)	15.9 (7.9-25.8)
3	Total Zn ($\mu\text{g g}^{-1}$)	15.5 (10.1-20.1)	16.2 (10.2-19.8)
4	Total Cu ($\mu\text{g g}^{-1}$)	31.0 (23.5-50.7)	19.4 (10.5-37.7)

Micronutrients (Total Fe, Mn, Zn and Cu) content in mango fruits

The data related to primary and secondary nutrient content in mango fruits at harvest from orchard 1 and 2 are presented in **Table 4.5** and depicted in **Fig.4.6**

As seen from the **Table 4.6** it was observed that the total iron, manganese, zinc and copper contents of mango fruits from orchard 1 were 10.8 to 77.7 with a mean value of $42.3 \mu\text{g g}^{-1}$, 10.5 to 29.5 with a mean value of $16.3 \mu\text{g g}^{-1}$, 10.1 to

20.1 with a mean value of $15.5 \mu\text{g g}^{-1}$ and 23.5 to 50.7 with a mean value of $31.0 \mu\text{g g}^{-1}$ respectively.

The data presented in **Table 4.6** showed that the total Iron, Manganese, Zinc and Copper contents of mango fruits from orchard 2 were 17.5 to 73.7 with a mean value of $38.3 \mu\text{g g}^{-1}$, 7.9 to 25.8 with a mean value of $15.9 \mu\text{g g}^{-1}$, 10.2 to 19.8 with a mean value of 16.2 and 10.5 to 37.7 with a mean value of $19.4 \mu\text{g g}^{-1}$ respectively.

Somewhat similar values of Fe, Mn, Zn and Cu were reported by Thakare (2016), Puranik (2015), Joshi (2015) and Patil *et al.* (2010).

CHAPTER V

SUMMARY AND CONCLUSION

5.1 Summary

The present investigation was undertaken to study the insecticidal residues in fruits of Alphonso mango. During investigation, an attempt was made to compare the insecticidal residue from the orchard which is located at Department of Agronomy, College of Agriculture, Dapoli (Orchard 1) and the orchard belonging to the private farmer (orchard 2). Persistence of insecticides recommended by Dr. B.S.K.K.V. Dapoli for the management of mango hopper complex was evaluated. Also, primary, secondary and micronutrient content of mango fruit samples from both the locations were analysed to know the total nutrient status of mango fruits at harvest stage.

The pivotal findings from present investigation are briefly summarised and concluded in this chapter.

5.1 Persistence of insecticides in mango fruits from orchard 1

Cypermethrin

The persistence of cypermethrin was tested at the spray concentration of 0.0075%. Its residue in mango fruit samples at egg stage was 0.06 ppm that reduced to 0.05 ppm at harvest stage which were below detectable level of 0.2 ppm

Quinolphos

The persistence of quinolphos was tested at the spray concentration of 0.05 per cent. Its residue in mango fruit samples at egg stage was 0.07 ppm which decreased upto 0.05 ppm at harvest stage which were below detectable level of 0.2 ppm.

Imidacloprid

The persistence of imidacloprid was tested at the spray concentration of 0.0053 per cent. Its residue in mango fruit samples at egg stage was 0.01ppm which is below detectable level of 1 ppm which further reduced to non detectable level at harvest stage of mango fruits.

Thiomethaxam

The persistence of thiomethaxam was tested at the spray concentration of 0.0025 per cent. Its residue in mango fruit samples was not detected in mango fruit samples at egg stage as well as at harvest stage

5.3 Insecticidal residue in mango fruit samples at harvest from orchard 1

Cypermethrin

Cypermethrin 25 EC was evaluated for its residue in harvest stage mango fruits. It was tested at the spray concentration of 0.0075 per cent as a recommended dose. It is evident from the result that the residual value of cypermethrin at 0.0075 per cent concentration in mango fruit samples was 0.05 ppm which was below detectable level of 0.2 ppm.

Quinolphos

Quinolphos 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the spray concentration of 0.05 per cent as a recommended dose. It is evident from the data that mean residual value of quinolphos at 0.05 per cent was 0.05 ppm which was below detectable level of 0.2 ppm.

Imidacloprid

Imidacloprid 17.8 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the spray concentration of 0.0053 per cent as a recommended dose. The results showed that spraying of imidacloprid at 0.0053 per cent concentration did not show any detectable residue in mango fruit samples at harvest stage.

Thiomethaxam

Thiomethaxam 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the spray concentration of 0.0025 per cent as a recommended dose. Spraying of thiomethaxam at 0.0025 per cent concentration did not show any detectable residue in mango fruit samples at harvest stage

5.3 Insecticidal residue in mango fruit samples at harvest from orchard 2

Cypermethrin

Cypermethrin 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the concentration of 1 ml L⁻¹ of water. The results showed that, the mean residual value of cypermethrin at the spray concentration of 1 ml L⁻¹ was 0.05 ppm which was below detectable level of 0.2 ppm.

Thiomethaxam

Thiomethaxam 25 EC was evaluated for residues in harvest stage of mango fruits. It was tested at the concentration of 0.4 g L⁻¹ of water. It is evident from the results that spraying of thiomethaxam at the dose of 0.4 g L⁻¹ of water did not show any detectable residues at harvest.

5.4 Comparison between insecticidal residue in mango fruits at harvest from orchard 1 and 2

It is evident from the results that cypermethrin and thiomethaxam were used in higher concentration in orchard 2 as compared to orchard 1. inspite of having higher dose, cypermethrin residues were found below detectable limit of 0.2 ppm in both case. i.e. 0.05 ppm for orchard 1 and 0.05 ppm for orchard 2. While thiomethaxam residues were not detected in any fruit samples from both mango orchards. It may be due to longer period of gap between spraying of insecticides and collection of fruit samples.

5.5 Primary, secondary and micronutrient content in mango fruit samples from orchard 1 and 2 at harvest stage.

Primary nutrient (Total N, P and K) content in mango fruits

The total nitrogen, phosphorus and potassium of harvest stage fruits as 0.06 to 1.18 with a mean of 0.55 per cent N, 0.11 to 0.29 with a mean value of 0.19 per cent P and 0.42 to 0.65 with a mean value of 0.52 per cent K in orchard 1

In orchard 2, the total nitrogen, phosphorus and potassium of harvest stage fruits were 0.03 to 1.10 with a mean value of 0.49 per cent N, 0.14 to 0.23 with a mean value of 0.17 per cent P and 0.42 to 0.77 with a mean value of 0.60 per cent K.

Secondary nutrients (total Ca, Mg and S) content in mango fruits

For orchard 1, the total calcium, magnesium and sulphur content in mango fruits ranged from 0.24 to 0.52 with the mean of 0.33 per cent Ca, 0.04 to 0.28 with a mean value of 0.12 per cent Mg and 0.21 to 0.61 with a mean value of 0.43 per cent S.

For orchard 2, the total Calcium, Magnesium and Sulphur content of mango ranged from 0.14 to 0.39 with a mean value of 0.28 per cent Ca, 0.02 to 0.26 with a mean value of 0.10 per cent Mg and 0.2 to 0.47 with a mean value of 0.37 per cent

Micronutrients (Total Fe, Mn, Zn and Cu)

It was observed that contents of total iron, manganese, zinc and copper contents of mango fruits from orchard 1 were 10.8 to 77.7 with a mean value of 42.3 $\mu\text{g g}^{-1}$, 10.5 to 29.5 with a mean value of 16.3 $\mu\text{g g}^{-1}$, 10.1 to 20.1 with a mean value of 15.5 $\mu\text{g g}^{-1}$ and 23.5 to 50.7 with a mean value of 31.0 $\mu\text{g g}^{-1}$ respectively.

The total Iron, Manganese, Zinc and Copper Contents of mango fruits from orchard no.2 were 17.5 to 73.7 with a mean value of 38.3 $\mu\text{g g}^{-1}$, 7.9 to 25.8 with a mean value of 15.9 $\mu\text{g g}^{-1}$, 10.2 to 19.8 with a mean value of 16.2 and 10.5 to 37.7 with a mean value of 19.4 $\mu\text{g g}^{-1}$ respectively.

Conclusion:

During present investigation two mango orchards were brought under study. The insecticides Viz. cypermethrin, quinolphos, imidacloprid and thiomethaxam were used in orchard 1 at the concentrations recommended by Dr.B.S.K.K.V.Dapoli. While cypermethrin and thiomethaxam were used by private farmer in orchard 2 at the concentration which is higher than recommended dose.

The present study indicated that the plant protection schedule recommended by Dr. Balasaheb Sawant Konkan Krishi Vidyapeeth, Dapoli is safe from the insecticidal residue point of view along with better plant protection. Eventhough the private farmer has used higher concentrations of insecticides the mango fruits were free from any residue due to longer interval. Hence, both the concentrations and time of insecticidal spray are important for obtaining residue free mango fruits.

The study of total nutrient content of mango fruit samples from both orchards revealed that the total N, P, Ca, Mg, S and micronutrients viz. Fe, Mn, Zn, Cu were higher in orchard 1 than orchard 2 except total K.

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APPENDIX – I
ABBREVIATIONS USED

%	:	Per cent
N	:	Nitrogen
P	:	Phosphorus
K	:	Potassium
Ca	:	Calcium
Mg	:	Magnesium
S	:	Sulphur
Fe	:	Iron
Mn	:	Manganese
Zn	:	Zinc
Cu	:	Copper
<i>et al.</i>	:	and others
Fig.	:	Figure
g	:	gram
mg	:	milligram
µg	:	Microgram
kg	:	Kilogram
a.i.	:	Active ingredient
ha	:	Hectre
mg kg ⁻¹	:	Milligram per kilogram
g a.i.ha ⁻¹	:	Gram active ingredients per hectre
ppm	:	Parts Per Million
a.i. kg ⁻¹	:	Active ingredients per

	:	kilogram
ml	:	Millilitre
ml kg ⁻¹	:	Millilitre per kilogram
lit	:	litre
ml L ⁻¹	:	Millilitre per litre
BDL	:	Below Detectable Limit
MRL	:	Maximum Residue Limit
ND	:	Not Detected
Viz.	:	Namely
@	:	At the rate
µm	:	Micrometer
mm	:	Millimeter
min	:	Minute
°C	:	Degree centigrade
hr	:	hour
µl	:	Microlitre
EC	:	Emulsifiable Concentration
EPA	:	Environmental Protection Agency
FAO	:	Food and Agricultural Organization
RT	:	Retention Time
SL	:	Soluble liquid
WP	:	Wettable powder
MT	:	Metric tonne
AF	:	Aqueous Flowable
WS	:	Water Soluble

FS	:	Flowable Concentrate
OD	:	Oil Dispersion
No.	:	Number
Fig.	:	Figure
GC	:	Gas Chromatography
MSD	:	Mass Selective Detector
UPLC	:	Ultra Performance Liquid Chromatography
MS	:	Mass Spectrometry



Chopped mango sample



Ethyl acetate extract of mango fruits

**PLATE-II: FINALLY CHOPPED MANGO FRUIT SAMPLES
AND THEIR EXTRACT**



Pre-flowering stage



Full flowering Stage



Egg stage



Harvesting stage

Plate-I: DIFFERENT GROWTH STAGES OF MANGO

Fig.4.1 Chromatograms for Cypermethrin

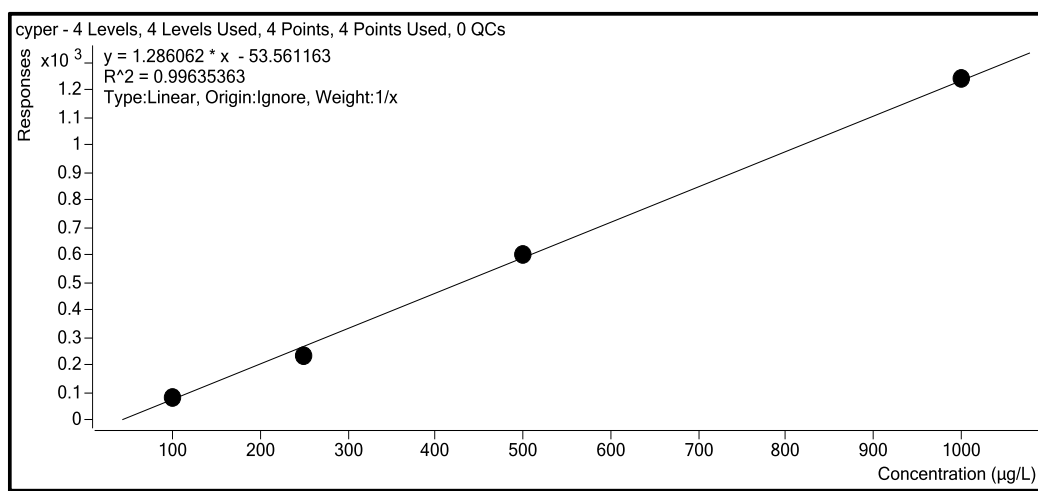


Fig.4.1.1 Calibration curve of cypermethrin standard

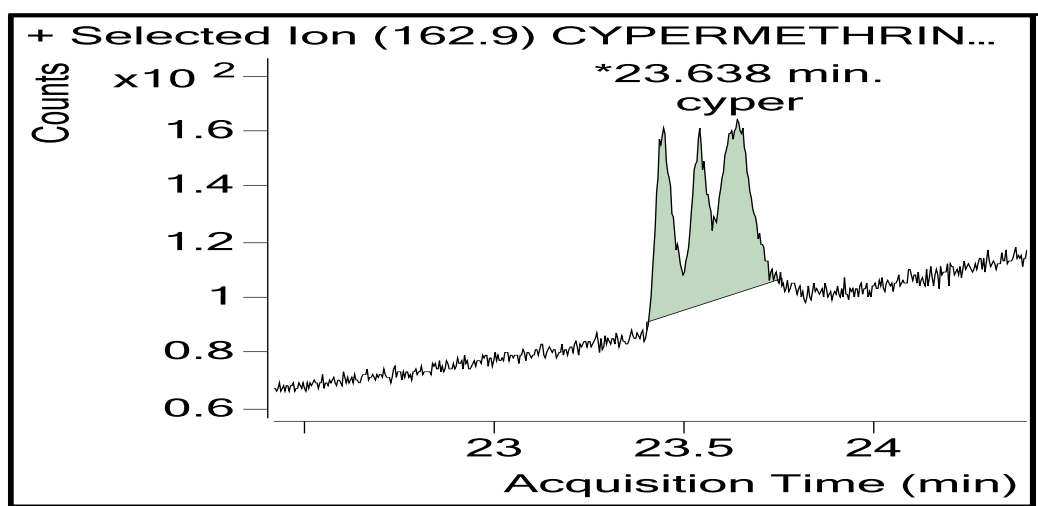


Fig. 4.1.2 One major eluted peak (at 23.64 min) of cypermethrin standard

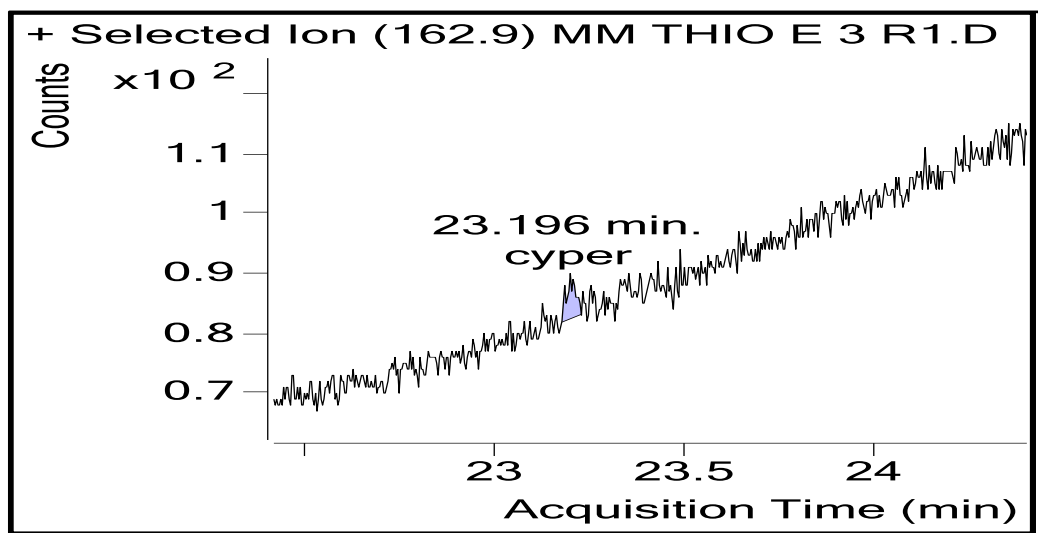


Fig.4.1.3 Chromatogram of mango fruit samples for cypermethrin at egg satge from orchard 1

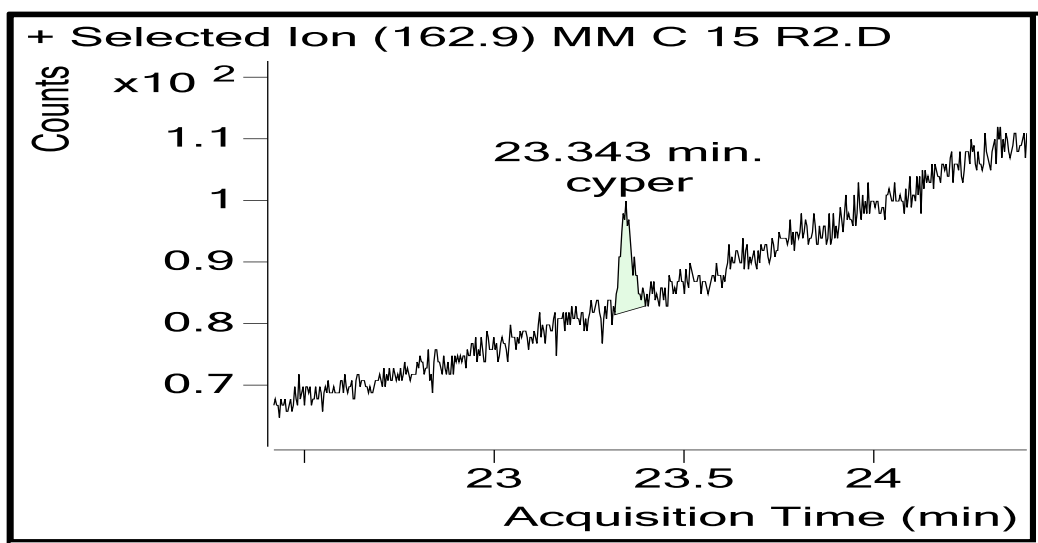
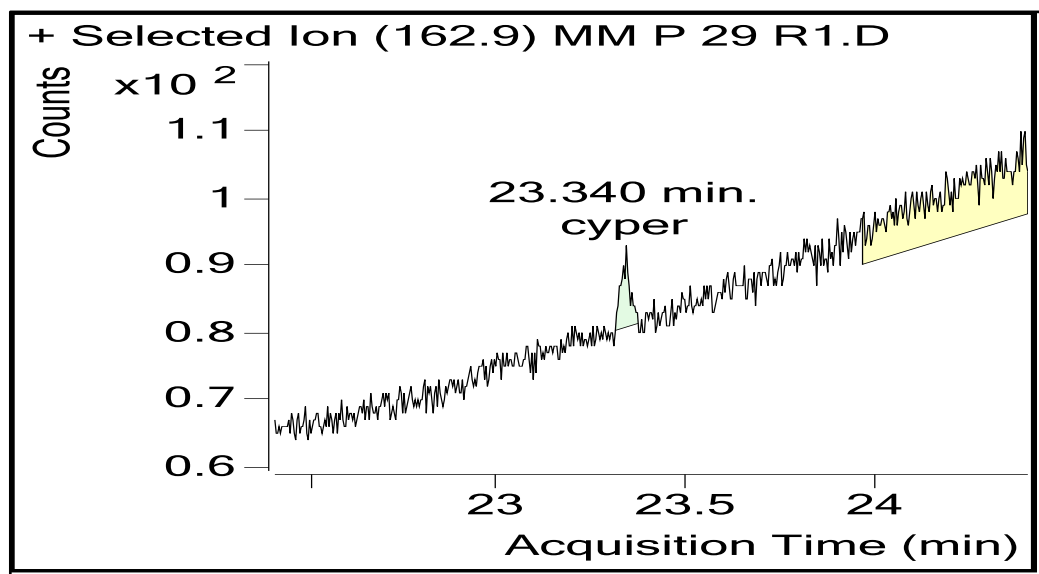


Fig.4.1.4 Chromatogram of mango fruit sample for cypermethrin at harvest stage from orchard 1



Fig, 4.1.5 Chromatogram of mango fruit sample for cypermethrin at harvest stage from orchard 2

Fig. 4.2 Chromatograms for Quinolphos

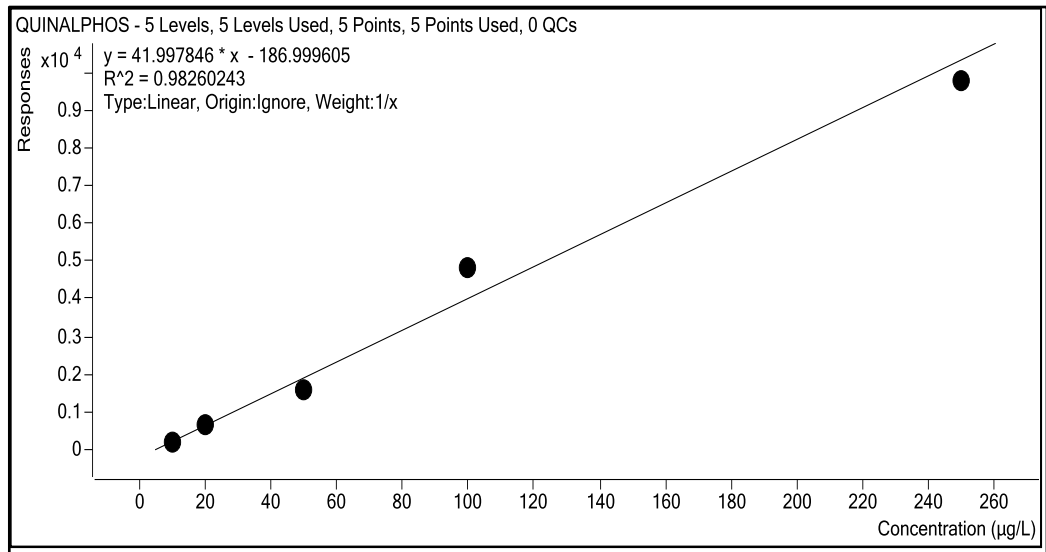


Fig. 4.2.1 Calibration curve of Quinolphos

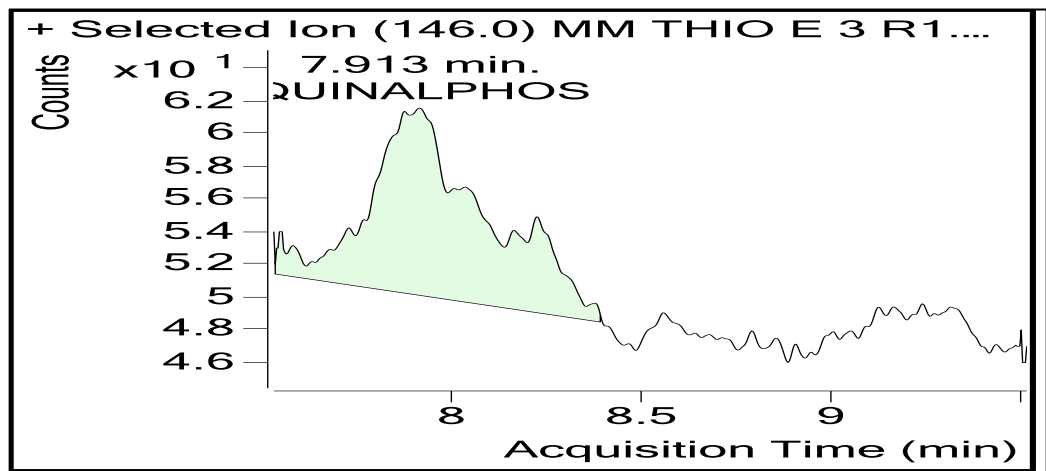


Fig.4.2.2 Chromatogram of mango fruit sample for quinolphos at egg stage from orchard 1

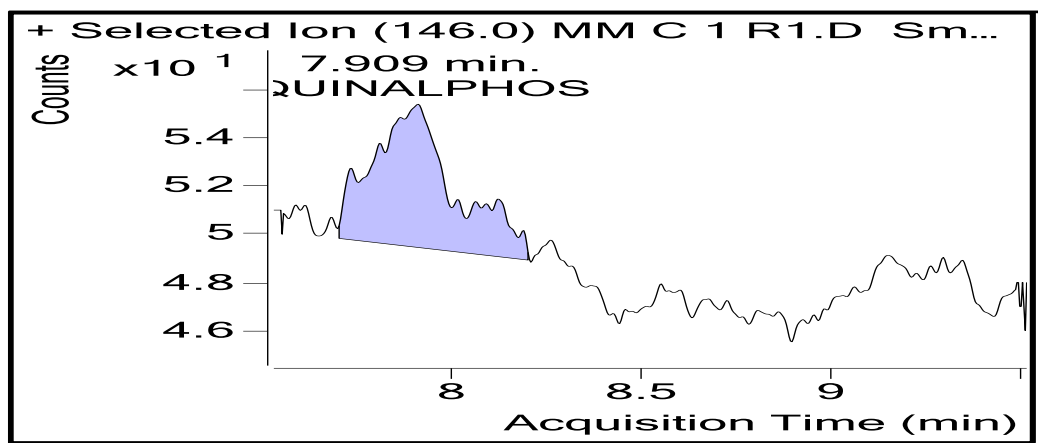


Fig. 4.2.3 Chromatogram of mango fruit sample for quinolphos at harvest stage from orchard 1

Fig. 4.3 Chromatograms for Imidacloprid

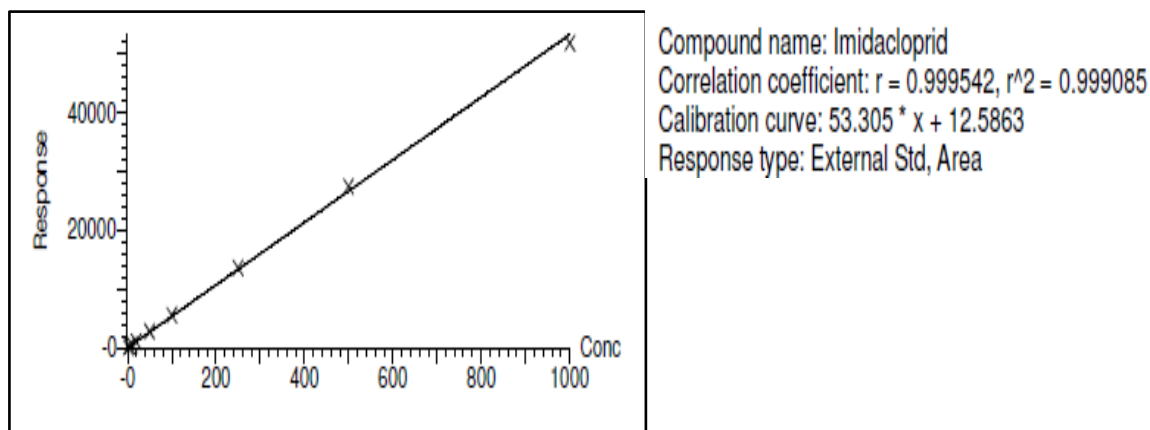


Fig. 4.3.1 Calibration curve of imidacloprid

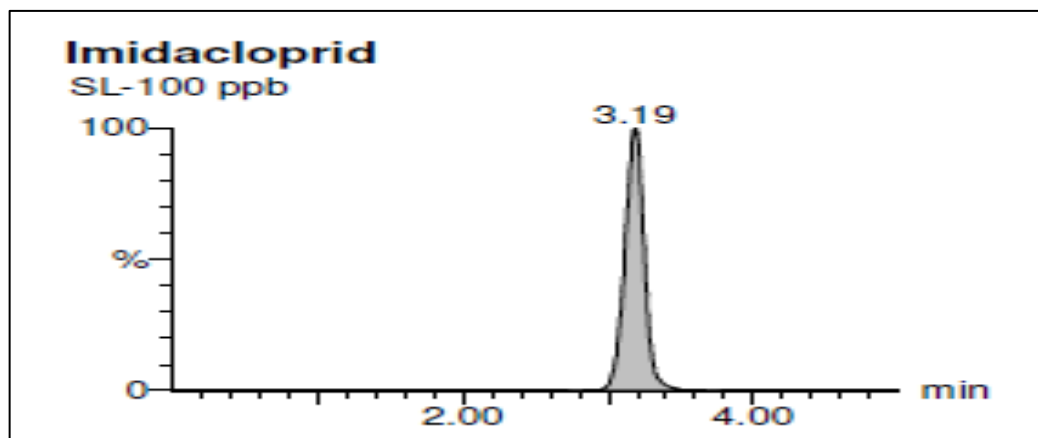


Fig.4.3.2 One major eluted peak (at 3.19 min) showed imidacloprid elution

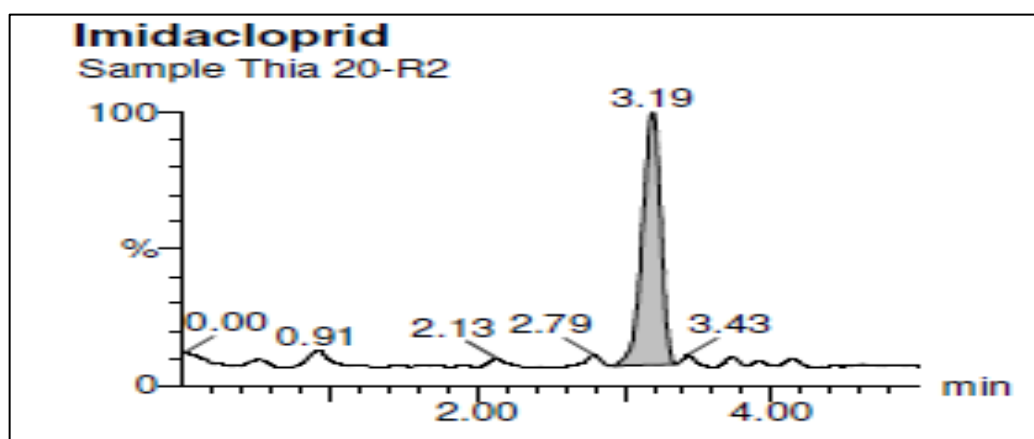


Fig. 4.3.3 Chromatogram of mango fruit sample at egg stage from orchard 1

Fig.4.4 Chromatograms for Thiomethaxam

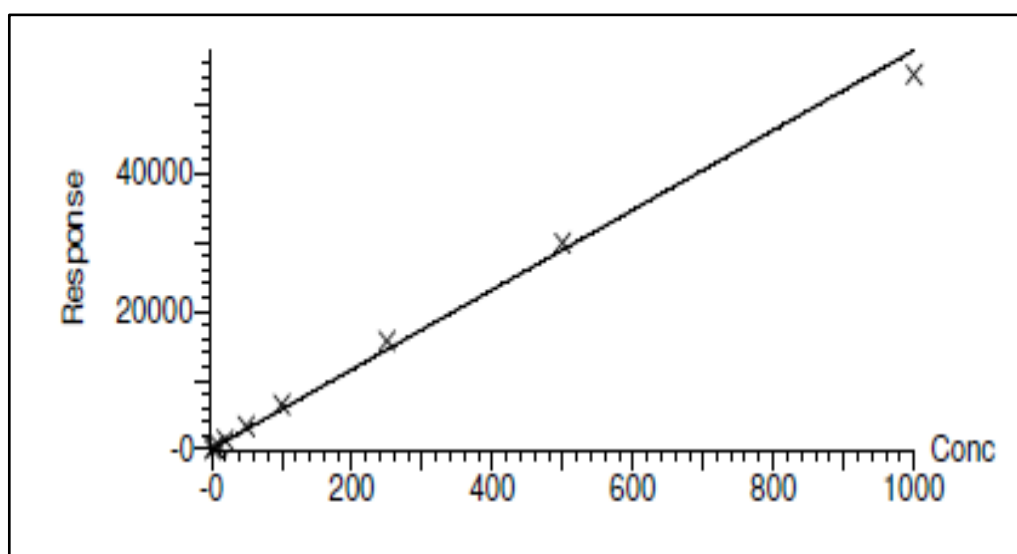


Fig. 4.4.1 Calibration curve of thiomethaxam

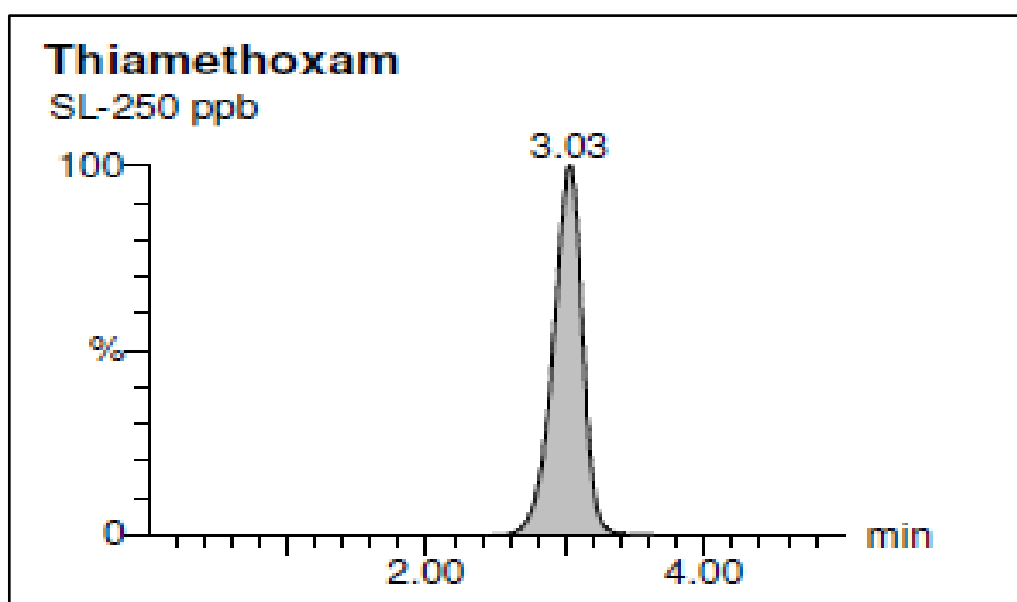


Fig. 4.4.2 One major eluted peak showed thiomethaxam elution

Fig.4.5 Macronutrient content of mango fruit samples from orchard 1 and 2

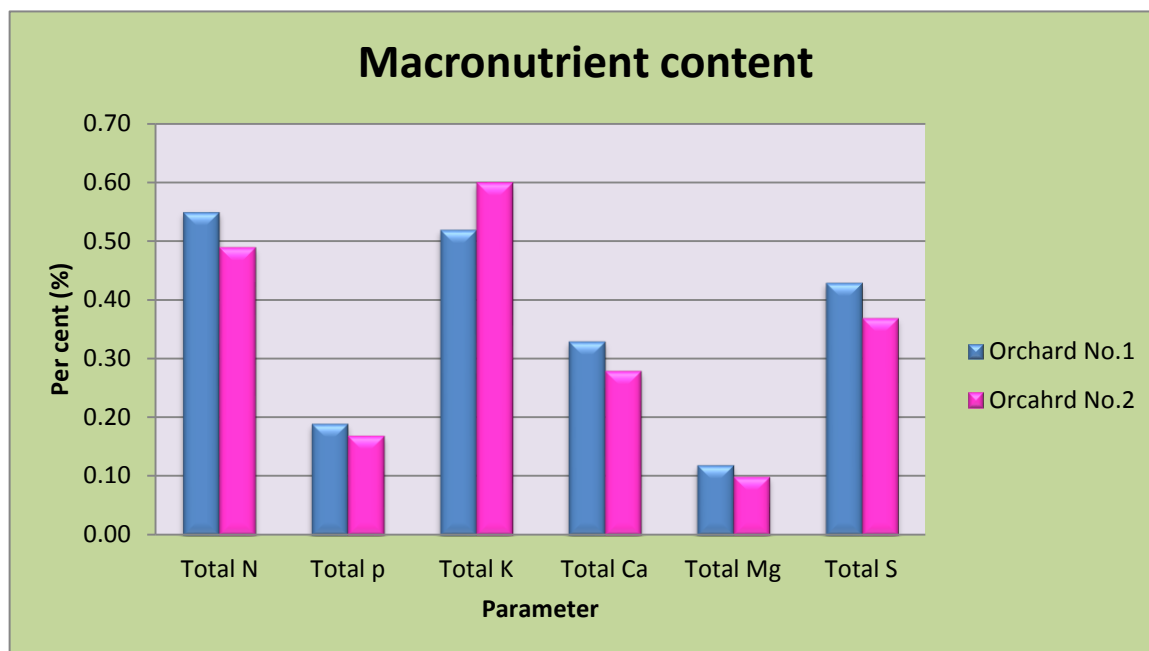


Fig. 4.6 Micronutrient content of mango fruit samples from orchard 1 and 2

