

## BIOEFFICACY AND SAFETY EVALUATION OF BIORATIONAL INSECTICIDES FOR THE MANAGEMENT OF SUCKING PEST COMPLEX OF CHILLI (CAPSICUM ANNUUM L.)

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### DECLARATION

I hereby declare that this thesis entitled "Bioefficacy and safety evaluation of biorational insecticides for the management of sucking pest complex of chilli (*Capsicum annuum* L.)" is a bonafide record of research work done by me during the course of research and the thesis has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar title of any other university or society.

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#### CERTIFICATE

Certified that this thesis entitled "Bioefficacy and safety evaluation of biorational insecticides for the management of sucking pest complex of chilli (*Capsicum annuum* L.)" is a record of research work done independently by Mrs. Thania Sara Varghese (2007-21-101) under my guidance and supervision and that it has not previously formed the basis for the award of any degree, fellowship or associateship to her.

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(Thania Sara Varghese)

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### LIST OF ABBREVIATIONS

%	Per Cent
/m <sup>2</sup>	Per sqare metre
<i>@</i>	At the rate of
a.i.	Active Ingredient
a.i. BDL	Below detectable level
CRM	Certified reference material
C.D.	Critical Difference
cm	Centimetre(s)
DAS	Days after sowing
DAT	Days after treatment
EC	Emulsifiable Concentrate
et al	And others
Fig.	Figure
g	Gram
h	Hour
ha <sup>-1</sup>	per hectare
kg	Kilogram
LCI	Leaf Curl Index
m	Metre
ml	Millilitre
mm	Millimetre
MRL	Maximum Residue Limit
OD	Oil Dispersion
PPM	Parts per million
RSD	Relative Standard deviation
SC	Suspension Concentrate
SD	Standard deviation
SL	Soluble Liquid
Spp	Species
Viz.	namely
	-

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# INTRODUCTION

#### 1. INTRODUCTION

Chilli (*Capsicum annuum* L.) is an indispensable spice used as basic ingredient in everyday cuisine all over the world. Chilli is said to have originated in the Latin American regions of the New Mexico and Gautemala as a wild crop around 7500 BC. The chilli crop came to the Asian continent as late as the 16<sup>th</sup> century with the identification of new sea routes by Portuguese and Spanish explorers. The south Asian climate suited to this vegetable crop, and since then a large percentage of chilli production has shifted to Asia. In the Indian subcontinent, chillies are produced through out the year. Two crops are raised in an year, during dry and wet season in the country. India is the largest producer and consumer of chillies in the world with a contribution of nearly 25 per cent of the global output. Trends in area, production and productivity showed an increase from 1950-51 to 2009-2010. The average production of chilli in India during 2009 is estimated to be around 11.67 lakh tonnes from 7.5 lakh hectares which contributed a share of 50-60 per cent share in global production (Reddy, 2010).

Andhra Pradesh stands first in the list of chilli producing states in India and has the maximum acreages and commands 49 per cent of the chilli production. The other major chilli producing states in India are Karnataka, Maharashtra, Orissa, Rajasthan and Tamil Nadu (Subbiah and Jeyakumar, 2009).

Although India's share in global chilli production is high, the productivity of chilli is only 1500kg ha<sup>-1</sup> which has to be increased to a bench mark level of 5000kg/ha to compete in the international market (Reddy, 2010). Among the different constraints that add up to low chilli productivity, the pest complex that attack chilli at different crop stages is most important. The pest spectrum of chilli is complex with more than 293 insects and a mite species debilitating the crop in the field as well as in storage (Asian Vegetable Research and Development Centre., 1987). A total of 39 genera and 51 species of pests were recorded by Reddy and Puttuswamy (1983 and 1984).

The major sucking insects that attack chilli are aphids (*Myzus persicae* Sulzer, *Aphis gossypii* Glover), mites (*Polyphagotarsonemus latus* Banks) and thrips (*Scirtothrips dorsalis* Hood). The yield loss due to chilli thrips and mites is estimated to the tune of 50 per cent (Ahmed *et al.*, 1987; Kandasamy *et al.*, 1990). The yield loss due to chilli mite may go up to 96.39 per cent (Borah, 1987) leading sometimes to complete failure of crop itself (Kulkarni, 1922). Chilli thrips multiply appreciably at a faster rate during dry weather periods and cause a yield loss of 30-50 per cent in South India (Varadharajan, 1994).

Due to monoculture of chilli, the pest build up is so much that farmers have to resort to a minimum of 5 to 6 chemical sprays. The number of sprays has increased over the years, but in vain and on the contrary, cost of cultivation has increased enormously making cultivation of chilli highly risky and non-profitable.

Crop pest reduce global food output by about 25 to 35 per cent and remain as the major barrier in international trade of agricultural produce. These high losses due to insects attacking crops, transmitting disease and inhibiting trade occur in spite of pesticide applications in agriculture. In 1995, world pesticide consumption reached 2.6 million metric tons of active ingredients, costing about US\$32 billion annually. The World Bank report on the environment revealed that the pesticide sales are on a strong upswing, and many highly toxic insecticides remain popular in developing countries (Koul et al., 2008). In spite of Integrated Pest Management successes in reducing pesticide use in some crops such as rice, a significant overall increase in pesticide use is likely over the next decade at least, with pesticide sales growing at a rate of about 5 per cent per year in India, China or Brazil. Pesticide use is particularly intense in export crops such as cotton, coffee, fruits, vegetables and cut flowers. The major class of insecticides which were very popular in insect control in developing countries till this time comprises organochlorines, organophosphates, carbamates and synthetic pyrethroids. The old insecticide chemistries (especially organophosphates and carbamates) are nerve poisons. Since, insects and other animals have similar

tissue, reproductive, hormonal and nervous systems, these compounds have potential for non target effects. This commonality made old insecticides highly toxic to non-target organisms including humans. Increased and non judicious use of non selective pesticides resulted in resistant development and resurgence of pests. Further, public concerns have grown considerably as a result of widespread environmental pollution, contamination of groundwater, and the presence of residues in food and water.

In view of this unsustainable reliance on chemical pesticides and public concerns, new and more environment-friendly techniques, approaches and strategies in the war against insect pests are called for. The rise of new generation of insecticides reduces the concern of ill effects of conventional insecticides. The new insecticide chemistries are more tissue-specific, highlybranched long-chain molecules that are activated in unique ways inside the target cells of insects resulting in reduced threat to other organisms. Selective toxicity to insects and safety to natural enemies have made the new class of insecticides more user and eco-friendly.

Residues in chillies have been reported by various workers in India (Nandihalli, 1979; Awasthi *et al.*, 2001; Dhotre *et al.*, 2001 and Joia *et al.*, 2001). There are reports of chilli consignment rejection from India in European countries due to the presence pesticide residues of ethion, triazophos, chlorpyriphos, phosphamidon, cypermethrin, fenvalerate and dicofol (Sreenivasa Rao, 2005). India contributes one fourth of the total quantity of chilli exported in the world followed by China which has been posing a severe competition to the Indian exporters. Major destinations of Indian exports are countries in West Asia, Far East, USA, Sri Lanka and Bangladesh. The exports can be further improved, provided India is able to meet the strict quality demands of international market.

The problem of insecticide residues in crop produces can be very well managed by observing proper waiting periods of insecticides on chilli crop. As

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the chilli fruits are harvested in 5- 6 days interval, if proper pre – harvest intervals were not observed residue will be a problem in harvested fruits. Chilli being consumed more as dried products rather than green chillies, the residue accumulation will be more on dry weight basis.

The World Health Organisation (WHO) estimate is that two million people get poisoned by pesticides annually and more than 50% of Indian food products are contaminated with pesticide residue (Roshini, 2003). Pesticide exposures are multiple, cumulative and may be additive or synergistic in effect. Children tend to be more exposed to toxic substances such as pesticides because they drink, eat and breathe more than adults. These residues of pesticides can also allegedly interfere with male and female hormones, causing infertility besides the carcinogenic and teratogenic effects caused by them which are suspected to be trans generational. At this juncture, we should make sure the safety of fruits and vegetables we consume in our daily routine. This is made possible to a large extent by some of the simple domestic decontamination procedures which can reduce the pesticide load in raw agricultural commodities to a larger extent.

With this background, a detailed study was undertaken with the following objectives:

- 1. To evaluate biorational insecticides for the management of sucking pest complex of chilli.
- 2. To assess the safety of selected insecticides to natural enemies.
- 3. To determine persistence and degradation of selected insecticides on chilli fruits.
- 4. To develop methods to decontaminate residues.

# **REVIEW OF LITERATURE**

#### 2. REVIEW OF LITERATURE

The literatures pertaining to the efficacy of insecticides for the management of sucking pest complex *viz.*, mite, thrips and aphids of chilli and the dissipation kinetics of selected insecticides on chilli fruits under the field conditions is reviewed and presented here. The reviews related to the safety/ toxicity of insecticides to the natural enemies of the sucking pests and non target biota too are detailed here. The literatures regarding the standardization of domestic practices to decontaminate residues from chilli fruit is also presented.

#### 2.1 SUCKING PESTS OF CHILLI

As per the results of the surveys conducted by A.V.R.D.C (Asian Vegetable Research and Development Centre) in Asia, the major sucking pests of chilli are mites (*Polyphagotarsonemus latus* Banks), thrips (*Scirtothrips dorsalis* Hood) and aphids (*Myzus persicae* Sulzer, *Aphis gossypii* Glover), (Berke and Shieh, 2000).

#### 2.1.1 Mite

Chilli mite, *P. latus* is a tiny spider like creature that is found in large numbers on the underside of leaves, covered with fine webs. Both nymphs and adults suck the cell sap and devitalize the plants. (Butani, 1976). Feeding of *P. latus* caused different types of physical deformities like thickening, brittleness and shortening, twisting, downward curling (Plate 1) and crumpling of young leaves. Midrib of young infested leaves bent in a zigzag fashion, ventral surface became silvery, petiole of mature leaves elongated and the plant became stunted with rosette symptoms. Infestation at flowering stage caused falling of flower bud. (Karmakar, 1997).

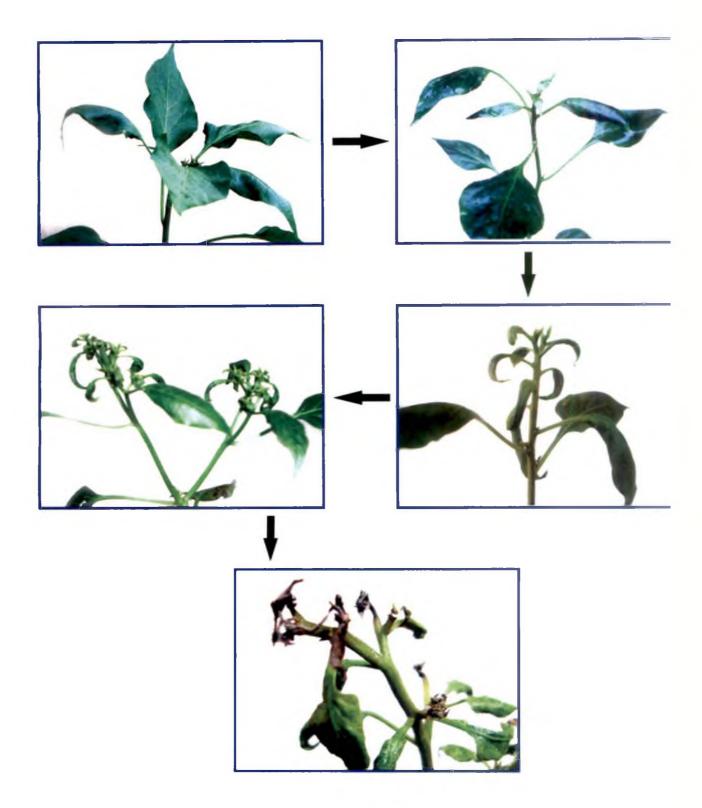


Plate 1. Different stages of Polyphagotarsonemus latus infestation in chilli plants

#### 2.1.2 Thrips

Chilli thrips, *S. dorsalis* is a polyphagous pest with a wide range of host plants. Both nymphs and adults lacerate the leaf tissue and suck the oozing juice, sometimes even the buds and flowers are attacked. Tender leaves and growing shoots are preferred, older leaves are seldom attacked. The infested leaves start curling upwards (Plate 2 (a)), crumple whereas buds become brittle and drop down. Pest infestation increases in dry weather. If there are no rains or sufficient irrigation systems, the entire plant may wither and dry away (Butani, 1976).

#### 2.1.3 Aphids

The cotton aphid *A. gossypii* and the peach green aphid, *M. persicae* are commonly found infesting chillies. Small, ovate, soft greenish brown sluggish nymphs and adults are found in large colonies (Plate 2 (b)) on the under surface of leaves and growing shoots of plants, sucking the cell sap. The aphids also secrete honey dew on which black sooty mould develops covering the leaves and twigs. This black coating hinders the photosynthetic activity of the plant causing further retardation in growth and fruiting capacity of the plant. (Butani, 1976).

#### 2.1.4 Murda syndrome

Chilli leaf curl symptom called murda is one of the most destructive syndromes affecting chilli in India and is considered to be caused by both mites and thrips (Puttarudraiah, 1959). The yield loss due to these two pests was estimated to be to the tune of 50 per cent (Ahmed *et al.*, 1987; Kandasamy *et al.*, 1990). The yield loss due to chilli mite may go up to 96.39 per cent (Borah, 1987) leading sometimes to complete failure of crop itself (Kulkarni, 1922). Chilli thrips multiplied appreciably at a faster rate during dry weather periods and causes a yield loss of 30-50 per cent in South India (Varadharajan, 1994).



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Plate 2. Chili Plants infested by (A) *Scirtothrips dorsalis* and (B)*Aphis gossypii* 

#### INSECTICIDES REGISTERED FOR SUCKING PESTS OF CHILLI IN INDIA

Directorate of plant protection, quarantine and storage and the Central Insecticide Board and Registration Committee (C.I.B & R.C) under the Department of Agriculture and Co-operation, Ministry of Agriculture, Government of India has notified the following insecticides against the sucking pest of chilli in its official website (source : www.cibrc.nic.in/major uses/insecticides).

Sl No	Insecticides	Dosage g ha <sup>-1</sup>		
		Mite	Thrips	Aphids
	Buprofezin 25SC	75-150		
	Carbofuran 3G		1000	
ĺ	Carbosulfan 25EC			200
	Chlorfenapyr 10SC	75-100		
	Deltamethrin 2.8 EC	300		
	Dimethoate 30 EC	300		
	Emamectin benzoate 5 SG	10	10	
	Endosulfan 35 EC			140
	Endosulfan 4 DP			140-175
	Ethion 50 EC	750 -1000	750-1000	
	Fenazaquin 10 EC	125		
	Fenpropathrin	75-100	75-100	
	Fenpyroximate 5 EC	15-30		
	Fipronil 5 S		40-50	40-50
	Hexythiazox 5.45 SC	15-25		
	Imidacloprid 17.8 SL		25-50	25-50
	Lambda Cyhalothrin 5 EC	15g	15g	
	Methomyl		300g	
	Milbemectin 1 EC	3.25		
	Novaluron 10 EC	1		
	Oxydemeton Methyl 25 EC	500	250	
	Phorate 10 G	1000	1000	
	Phosalone 35 EC	450	700	1000
}	Propargite 57 EC	850		700
	Quinalphos 25 EC	375		
	Quinalphos 15 DP			250
	Spinosad 45 SC		73	300
	Spiromesifen 22.9 SC	96		
	Thiacloprid 21.7 SC		54-72	
	Indoxacarb + Acetamiprid		88.8-111	

Table 1. List of insecticides registered by C.I.B & R.C against chilli mites, thrips and aphids.

# 2.2 EFFICACY OF INSECTICDES AGAINST SUCKING PESTS OF CHILLI.

#### 2.2.1 Conventional insecticides

#### 2.2.1.1 Organophosphates

#### Dimethoate

Bagle (1998) reported the efficacy of dimethoate 0.03 per cent and 0.05 per cent when compared to neem product and other insecticides against thrips in chilli variety Pusa Jwala and G<sub>4</sub> and found that dimethoate 0.05 per cent was found superior over dimethoate 0.03 per cent. The superiority of dimethoate 0.03 per cent when compared to neem products in controlling chilli thrips and aphids was reported by Mallikarjuna Rao *et al.* (1999 a and b), where a single round of dimethoate 0.03 per cent spray and four sprays of phosalone 0.05 per cent gave good control of the sucking pest. Dimethoate 0.03 per cent sprayed crop had only 42.38 per cent leaf curl incidence due to chilli mite compared to methyl -o- demeton 0.025 per cent and water sprayed crop which recorded 51.65 per cent and 63.32 per cent respectively (Panickar and Patel, 2001). Dimethoate 30 EC caused 100 per cent mortality of *A. gossypii* on Okra. (Boopathi *et al.*, 2010).

#### Ethion

Gangopadhyay and Sarkar (2000) reported the overall performance of ethion (0.04%, 0.06%, and 0.08%) and dicofol (0.03%, 0.05%, and 0.07%) in reducing chilli mite than other treatments *viz.*, monocrotophos (0.03%, 0.05%, and 0.07%) and endosulfan (0.03%, 0.05%, and 0.07%). Ethion 50 per cent EC proved to be superior in controlling the leaf curl disease of chilli with 6.41 per cent incidence through suppression of chilli thrips and mite and recorded the highest yield of green chilli (Mishra, 2003). Sarangi and Panda, (2004) reported that carbosulfan, triazophos and ethion caused 81.00, 71.00 and

66.00 per cent reduction in chilli thrips population, respectively as compared to pre-spray population, when applied as foliar sprays.

Bioefficacy of certain insecticides were studied against chilli thrips at student's farm, College of Agriculture, Rajendranagar, Hyderabad of which fipronil 0.01% and triazophos 0.08% were most effective against chilli thrips followed by profenofos 0.10%, ethion 0.10% and cypermethrin 0.0012% (Mahalingappa *et al.*, 2008).

A field experiment on evaluation of some inorganic insecticides and botanicals for the control of chilli thrips at the main vegetable research station, Anand Agricultural University, Anand 2002-2003 and 2003 -2004 revealed that among the different insecticidal treatments, ethion + cypermethrin (0.045%) and methomyl (0.04%) proved to be the most effective treatments (Patel *et al.*, 2009).

#### Oxy- demeton- methyl

The effect of methyl-o-demeton 25 EC @ 500 ml/ha persisted for 15 days against chilli aphids and 17 days against leafhopper. (Ramesh babu and Santharam, 2000). The efficacy of methyl demeton 25EC @ 500ml ha<sup>-1</sup> and dimethoate 30 EC @ 500ml ha<sup>-1</sup> in controlling chilli thrips was reported by (Soundrarajan *et al.*, 2001). In an IPM module of chilli pests, it was suggested to have sprays of oxy- demeton methyl 0.05 per cent at 2-3 weeks interval at 3-4 weeks after planting against chilli thrips (Shukla, 2004). Among the five different pest management modules (M) evaluated for the management of thrips, *S. dorsalis* on sweet pepper under protected and open field conditions, acephate – oxy-demeton – methyl (M1) was effective in reducing thrips damage (Reddy and Kumar, 2006).

#### 2.2.2 New generation insecticides

#### 2.2.2.1 Neonicotinoids

Neonicotinoid insecticides act as agonists of the insect nicotinic acetylcholine receptor (nAChr) located in the central nervous system. They are effective against sucking insects such as aphids, whiteflies and plant hoppers, beetles and some lepidopteran pests and can be used as foliar sprays, seed treatment and soil application (Nauen and Bretschneider, 2002).

#### Imidacloprid

Jarande and Dethe (1994) reported that seed dressing with imidacloprid 70WS @ 15g per kg of seeds followed by root dip of seedlings with 0.03 per cent imidacloprid gave excellent control of sucking pests and also resulted in an yield of 63.47 q/ha green chillies as against 37.42 q/ha in untreated crop. Seed treatment with imidaclorprid also showed phytotonic effect on seedlings in nursery by showing maximum growth and higher total chlorophyll content of leaves.

Sunanda and Dethe (1998) reported that, nursery treatment of chilli seeds with imidacloprid 70 WS at the rate of 15g kg<sup>-1</sup> of seeds was effective in keeping the chilli seedlings free from sucking pests *viz.* aphids, thrips and mites. The effect of imidacloprid spray persisted for 23 days against aphids at 100 and 150 ml ha<sup>-1</sup> and 29 and 31 days against leafhopper at 100 and 150 ml ha<sup>-1</sup>, respectively in chilli ecosystem. (Ramesh babu and Santharam, 2000).

Plant hole treatment (PHT) with imidacloprid (0.5 g  $l^{-1}$ ) followed by acephate (1g  $l^{-1}$ ) spray at 70 days after planting effectively controlled mite and thrips. PHT alone with imidacloprid recorded lowest leaf curl index due to mite and thrips (Manjunatha *et al.*, 2000). In another study by the same authors, the treatment with imidacloprid @ 150 ml ha<sup>-1</sup> recorded significantly highest yield followed by imidacloprid @ 125 and 100 ml ha<sup>-1</sup>. Lowest leaf curl index in chilli due to thrips was recorded in the sequential treatment

which included seed dress and seedling dip with imidacloprid 75SP 0.5 g l<sup>-1</sup> (Manjunatha *et al.*, 2001). Patil *et al.*, (2002) reported that imidacloprid 17.8 SL @ 125 ml and 150 ml ha<sup>-1</sup> was highly effective against the important sucking pest complex of chilli and proved to be better than monocrotophos and dimethoate. The treatment with imidacloprid @ 150 ml ha<sup>-1</sup> recorded significantly highest yield followed by imidacloprid @ 125 & 100 ml ha<sup>-1</sup> monocrotophos 36 WSC @ 650 ml ha<sup>-1</sup> and dimethoate 30 EC @ 750ml ha<sup>-1</sup>.

In IPM packages for sustainable production of chilli, it is suggested to include new molecules (thiamethoxam, abamectin, diafenthiuron, spinosad, imidacloprid, acetamiprid, indoxacarb, fenazaquin, fipronil, propargite, and fenpropathrin) along with botanicals to protect the crop from pest complex (Noor, 2004).

Sarangi and Panda, (2004) reported the efficacy of imidacloprid, as Seedling Root Dip (SRD) in reducing thrips population in chilli. Imidacloprid 17.8 SL @ 250 ml ha<sup>-1</sup> was observed to provide reduction of the whitefly *Bemissia tabaci* Genn in chilli at 1, 3, 5, 7 and 14 days after sprays *i.e.* 89.86, 95.58, 81.50 and 58.98 per cent respectively, followed by acephate 75 SP at 1250 g ha<sup>-1</sup> and methyl demeton 25 EC at 1250 ml ha<sup>-1</sup> (Singh *et al.*, 2004a).

Imidacloprid 17.8 SL (0.022 kg a.i.  $ha^{-1}$ ) was the most effective in suppressing the thrips population (average of 1.46 thrips per 10 apical leaves) and increasing the pod yield of chilli (27.63 q  $ha^{-1}$ ), followed by monocrotophos and acetamiprid (Mishra *et al.*, 2005).

Field experiment conducted by Singh *et al.*, (2005) at Chattisgarh, India, showed that imidacloprid 17.8 SL at 200 ml ha<sup>-1</sup> was the most effective against *S.dorsalis* and *A. gossypii* in chilli cv. Pusa Sadabahar. The green chilli yield was also the highest from the plots applied with imidacloprid. Bhede *et al.*, (2008) reported that application of phosphamidon 40% + imidacloprid 2% SP was more effective for suppression of thrips population and also increased the yield of green chilli.

#### Acetamiprid

Acetamiprid 20 SP @ 80 and 40 g a.i. ha<sup>-1</sup> were effective in reducing the sucking pests of chillies followed by acetamiprid 20 SP @ 20 g a.i. ha<sup>-1</sup> and recorded maximum green chilli yield (Jayewar *et al.*, 2003). Fipronil 5 SL (0.01%) followed by thiamethoxam 25 WG (0.005%), acetamiprid 20 SP (0.002%) and dimethoate 30 EC (0.06%) were most effective against thrips, while carbaryl 50 WDP (0.15%) followed by phosalone 50 EC (0.07%), and chlorpyrifos 20 EC (0.05%) were least effective.(Reddy *et al.*,2005).

The treatments of indoxacarb14.5SC+ acetamiprid 7.7SC (RIL-042 222 SC) was significantly superior in reducing the incidence of sucking pest and fruit damage by *Helicoverpa armigera* (Hubner) followed by acetamiprid 20SP @ 200g ha<sup>-1</sup>. The indoxacarb14.5SC+ acetamiprid 7.7 SC combinations @ 500ml ha<sup>-1</sup> also registered the highest green chilli fruit yield (Dharne and Kabre, 2009). Among the different newer molecules, acetamiprid 20SP @ 200g ha<sup>-1</sup>, indoxacarb14.5SC @ 500ml ha<sup>-1</sup>, and a combination product of indoxacarb 14.5 SC + acetamiprid 7.7% SC @500ml ha<sup>-1</sup> were on par with each other in reducing thrips population in all the three sprays during 2005 and 2006 (Nandihalli, 2009).

#### Thiamethoxam

Thiamethoxam, a new class of thianicotinyl compound, was found to be very effective for the control of homopterans (Mohan and Katiyar, 2000). The sequential sprays of fipronil 5 SL (0.01%) followed by thiamethoxam 25 WG (0.005%) and dicofol 18.5 EC (0.09%) were found to be the most effective insecticides against the mites, while carbaryl, followed by dichlorvos 76 EC (0.076%), and indoxacarb14.5 SL (0.0145%), were the least effective against mites (Reddy et al., 2005).

A field experiment was conducted during kharif 1999 to 2001 to evaluate the different doses of thiamethoxam 25 WG and diafenthiuron 50 WP for controlling chilli thrips infesting chilli cv S - 49 cultivated in middle Gujarat. The crop protected with two higher doses of diafenthiuron (60 and 50 g a.i. ha<sup>-1</sup>) proved its superiority over lower doses (30 and 40 g a.i. ha<sup>-1</sup>) and all the doses of thiamethoxam (25.0, 37.5 and 50.0 g.a.i.ha<sup>-1</sup>) and was on par with the standard chemical checks *viz.*, monocrotophos (144 g a.i. ha<sup>-1)</sup> and triazophos (160 g a.i. ha<sup>-1</sup>) one day after spraying (Patel *et al.*, 2006). Studies were conducted to evaluate the efficacy of different seed treatment insecticides against *S. dorsalis* in chilli. Imidacloprid 75% WS at 5 and 8 g kg<sup>-1</sup> seed, acetamiprid 20% SP at 5 and 10 g kg<sup>-1</sup> seed and thiamethoxam 75% WS at 5 and 10 g kg<sup>-1</sup> seed were used as treatments. Among the treatments, thiamethoxam 75% WS at 10 g kg<sup>-1</sup> seed was significantly superior in efficacy against thrips up to 15days after sowing (DAS), followed by thiamethoxam 75% WS at 8 g kg<sup>-1</sup> seed. (Reddy *et al.*, 2006).

In a study conducted by Nagaraj *et al.*, (2007) the mean thrips population and leaf curl index (LCI) recorded in chilli were minimum in thiamethoxam 25 WG (35 g a.i.  $ha^{-1}$ ) treated plots which was followed by imidacloprid 17.8 SL (35 g a.i.  $ha^{-1}$ ), thiacloprid 21.7 SC (30 g a.i.  $ha^{-1}$ ) and clothianidin 50 WG (60 g a.i.  $ha^{-1}$ ).

#### 2.2.2.2 Microbial fermentation products

#### Spinosad

Spinosad is a naturalyte class of insecticide and primarily consist of two macro cyclic lactones Spinosyn A and D which are secondary metabolite produced by the actinomycete *Saccharopolyspora spinosa* under natural fermentation conditions (Salgado, 1997). Spinosad, imidacloprid, novaluron, abamectin

and spiromesifen were found equally effective in suppressing thrips population on 'Scotch Bonnet' varitety of chilli (pepper) in St.Vincent (Seal and Klassen, 2006). Field experiments were conducted on chilli crop to assess the bioefficacy of spinosad 45 SC on chilli thrips and pod borer *Spodoptera exigua* at Regional Agricultural Research station, Guntur. Spinosad 45 SC tested at 100, 125, 162.5 and 200 ml ha<sup>-1</sup> was effective against chilli thrips and pod borer. Spinosad 45 SC @ 125 ml ha<sup>-1</sup> exhibited efficacy equal to higher doses and was recorded to be the optimum dose for control of thrips and pod borer on chilli crop (Prasad and Ahamed , 2009).

#### Avermectins

Abamectin (Vertimec 1.8 EC; 5 and 9 g a.i. ha<sup>-1</sup>) at both dosages recorded the highest mortality (population reduction) of the moving stage as well as different life stages (male, female and larva) of the yellow mite (*P. latus*) in chilli (cv. Bullet), followed by fenazaquin (Magister 10 EC; 50 and 100 g a.i. ha<sup>-1</sup>). Clofentezine (Apollo 50 SC; 250 and 300 g a.i. ha<sup>-1</sup>) was the least effective acaricide (Sarkar *et al.*, 2005). Sarkar *et al.*, (2008) conducted a study to evaluate the effect of fenazaquin, abamectin and clofentizine in two different doses against yellow mite, *P. latus* in chilli. Amongst them, abamectin (Vertimec 1.8 EC), a GABA – ergic nerve poison was found to be the most suitable in both dosages (9 g and 5 g a.i. ha<sup>-1</sup>), followed by fenazaquin (Magister 10 EC), a METI group of chemical at 100 g a.i. ha<sup>-1</sup>.

Field experiments were conducted at Regional Agricultural Research Station, Guntur, Andhra Pradesh on chilli crop during 2002-03 and 2003-04 to test the bio efficacy of emamectin benzoate 5% SG on thrips, *S. dorsalis* and pod borer, *S. litura*. The pooled data revealed that carbaryl @ 750 g ai ha<sup>-1</sup> (3.88/leaf) and emamectin benzoate @ 10 g a.i. ha<sup>-1</sup> (4.71/leaf) were superior in managing thrips (Ahamed and Prasad, 2009)

#### 2.2.2.3 Oxadiazine derivative

#### Indoxacarb

Results of the field evaluation of the bioefficacy of certain new insecticides along with traditional insecticides as foliar sprays against chilli thrips and mites undertaken under irrigated conditions during 2001 and 2002, indicated that among the 17 insecticides tested, endosulfan 35 EC at 0.07%, quinalphos 25 EC at 0.055% and indoxacarb 14.5 SC at 0.0145% were found to be least effective against thrips and mite (Reddy *et al*, 2007a). The treatments of indoxacarb14.5 SC+ acetamiprid 7.7 SC (RIL-042 222 SC) was significantly superior in reducing the incidence of sucking pests viz. thrips, whiteflies and aphids in chilli. (Dharne and Kabre, 2009).

#### 2.2.2.4 Keto – Enase group

#### Spiromesifen

Spiromesifen, a spirocyclic phenyl substituted tetronic acid derivative is one such new insecticide possessing potential activity and novel mode of action. Spiromesifen at 72, 96 and 120 g a.i. ha<sup>-1</sup> recorded 83.8, 86.6 and 91.7% mean reduction of chilli mite population respectively. Spiromesifen showed better efficacy than dicofol. Among the different dosages applied, spiromesifen at higher dose, 120g.a.i.ha<sup>-1</sup> was superior in controlling chilli mite. But the lower dose of 72 g a.i.ha<sup>-1</sup> was economically viable, if applied at the early stage of mite infestation. Spiromesifen had a long lasting efficacy on chilli and at 120 g a.i.ha<sup>-1</sup>, reduced the leaf curl damage from 41.8 to 12.5%. Application of 240 g a.i.ha<sup>-1</sup> spiromesifen did not produce any phytotoxicity. (Kavitha *et al.*, 2006).

The lowest mean population of mite and its leaf curl index was recorded in spiromesifen 240 SC (8.17 mites/ leaf and 1.61 LCI) in a study at Agricultural College Farm Bheemarayanagudi during kharif 2006. Ethion 50 EC recorded 13.53 mites/ leaf and 2.41 LCI which were at par with dicofol

quinalphos > endosulfan > chlorpyrifos > clothianidin > ethion (Dhawan et al., 2008).

#### 2.2.3.2 Microbial Fermentation Products

#### Spinosad

In the plant pest notice published by Central Science Laboratary UK, it was recommended that spinosad was effective against *S. dorsalis* on ornamentals and cucumbers. (Collins *et al.*, 2006). Spinosad 45 SC a new A: D ratio (Spinosyn A 50% Min + Spinosyn D 50 % Max) at the rate of 75 g a.i ha<sup>-1</sup> was found to be effective and optimum to combat cotton thrips and bollworms (Bheemanna *et al.*, 2009).

#### Avermectins

In the plant pest notice published by Central Science Laboratary UK, it was recommended that abamectin was effective against *S. dorsalis* on ornamentals and cucumbers. (Collins *et al.*, 2006). Among the different chemicals assayed, abamectin 0.0009 % provided best control of *P. latus* infesting *Jatropha curcas* L (Kavitha *et al.*, 2007).

#### 2.2.3.2 Keto enase group

#### Spiromesifen

In a field trial conducted at Chandra Krishi Vishwavidyalaya, West Bengal, spiromesifen (Oberon-240SC) gave excellent control of red spider mite of brinjal along with significant increase in yield. (Sekh *et al.*, 2007). Studies on the bioefficacy of spiromesifen 240 SC conducted at instructional farm, RCA, MPUAT, Udaipur during summer and Kharif, 2009 revealed that two sprays of spiromesifen 240 SC at 625 ml/ha at 28 days interval was found most effective against mite and whitefly in tomato (Ameta *et al.*, 2010).

#### 2.3 SAFETY TO NATURAL ENEMIES AND POLLINATORS

#### 2.3.1 Conventional insecticides

#### **Organochlorines and Organophosphates**

Croft and Nelson (1972) evaluated toxicity of twenty three commonly used pesticides against *Amblyseius fallacies* and reported that dimethoate was less toxic compared to carbaryl and phosalone. Application of dimethoate (0.05%) was found to have some adverse effect on predatory insects (Nurindah and Bondra, 1988). Jagadish and Channabasavanna (1989) conducted toxicity studies on *A. tetranychivorus*, an effective predator *Tetranychus ludeni*, and was found that the predator was more susceptible to quinqlphos and malathion at 0.05% and 0.01% concentration followed by dicofol at 0.05% at 12 hours after treatment. According to Hegde and Patil (1994) dicofol 18.5 EC, monocrotophos 36 SL, wettable sulphur 80 WP and ethion 50EC caused 100 per cent mortality of *Amblyseius longispinosus* (Evans) after 24 hrs of spray in cotton ecosystem. Rai *et al* (1995) tested toxicity of pesticides including neem products on *Amblyseius alstoniae* Gupta and found that monocrotophos and dicofol were toxic.

Dimethoate at 250 g a.i. ha<sup>-1</sup> resulted in 100 per cent mortality of the parasitoid *Aphidius* spp within 24 hours. (Tonet *et al.*, 1997). At 0.07% concentration, dimethoate was highly toxic to *Aphytis melinus* DeBach and *Chiocorus nigrta* (Fabricius) (Krishnamoorthy and Rajagopal, 1998). The insecticide was also toxic to *Coccinella sexmaculata* (Thayaalini and Raveendranath, 1998) and showed high residual toxicity to *Cryptolaemus montrouzeieri* Mulsant (Sundari, 1998).

Of the five pesticides tested on *Chrysoperla carnea* (Stephens), monocrotophos, dicofol and endosulfan proved safer compared to dimethoate and malathion (Sharanabasava *et al.*, 1999). Foliar application of dimethoate 0.005% was more toxic to coccinellid, syrphid, spider and braconid population up to 10 days after spraying in okra (Thamilvel, 2004).

#### 2.3.2 New generation Insecticides

#### 2.3.2.1 Neonicotenoids

#### Imidacloprid

Imidacloprid had no effect on spiders (Xin and Xi, 1995). In plants treated with the insecticide, there was significant decrease in the general mobility of coccinellid predator, *Coliomegilla maculate* (De Geer) (Smith and Krischik, 1999). The toxicity of imidacloprid to the mirid predators viz., *Macrolophus calignosus* Wagner and *Dicyphus tamaninii* Wagner was reported (Figulus *et al.*, 1999). The seed dressing of cotton seeds with imidacloprid @10g/kg seeds found safer to *C. carnea* and ladybird beetles in the cotton ecosystem (Katole and Patil, 2000). Laboratory studies showed that imidacloprid at 0.07% was persistent up to 15 days and caused 24.7% mortality of *Coccinella sexmaculata* adults. (Patil and Lingappa, 2000). The green lace wing, *C. carnea* and imidacloprid 0.34 kg a.i. ha<sup>-1</sup> were compatible and they together controlled the white fly, *B. tabaci* resulting in the highest yield and moderate plant viral infection in tomato(Ruiz and Medina, 2001).

Imidacloprid 20 SL (0.004%) were safe to predators like cocinellids, spiders, and chrysopids as evidenced by the highest survival rate after the use of the insecticides under field condition (Chandrasekharan, 2001). Imidacloprid at 0.25% was safer to aphid predators *viz.*, *C. carnea* and *Menochilus sexmaculatus* and *Coccinella transversalis* than organophosphate insecticides like chlorpyriphos (0.05%), profenofos (0.05%) and triazophos (0.05%) on cowpea (Varghese, 2003). Imidacloprid has an inhibitory effect on honey bees (Medrzycki *et al.*, 2003).

The seed treatment of transgenic cotton with imidacloprid @ 5g/ kg of seed was found to be safe to the natural enemy population and it attracted

predators, viz. coccinellid beetles, green lace wings and different types of spiders (Kannan *et al.*, 2004). With the application of imidacloprid, the phytoseiid predator of chilli thrips *Euseius sojaensis*, was found to survive on grapes, whereas by the application of permethrin the predator population declined (Shibao *et al*, 2006).

The toxicity of synthetic insecticides such as imidacloprid (0.01%), acetamiprid (0.002%), thiamethoxam (0.02%), profenofos (0.05%) and carbosulfan (0.14%), were compared with monocrotophos (0.05%) for their toxicity to *Trichogramma chilonis* and *Cheilomenes sexmaculata*. Among the insecticides, carbosulfan was highly toxic to T. chilonis and C. sexmaculata, followed by acetamiprid. Imidacloprid was relatively less toxic to both species. (Basappa, 2007).

The coccinellid predator *Hippodamia undecimnotata* that fed upon *Aphis fabae* was reared on *Vicia faba* plants treated with the systemic carbofuran and imidacloprid. Survival of immature stages in insecticide treatments (67.6% and 52.2%, for carbofuran and imidacloprid, respectively) was lower than control (77.4%). Adult average longevity was significantly higher for the control than the insecticides treatments. Population increase parameters were also adversely affected by the application of insecticides. (Papachristos and Milonas, 2008).

#### Acetamiprid

In a study on the toxicity of newer molecules of insecticides, acetamiprid (20 g a.i. ha<sup>-1</sup>), thiamethoxam (25 g a.i. ha<sup>-1</sup>), NACLFMOA (20 g a.i. ha<sup>-1</sup>), abamectin (20 g a.i. ha<sup>-1</sup>), were found to be relatively safer to the predatory ladybird beetles(Acharya *et al.*, 2002).

Acetamiprid (0.001, 0.002, 0.003 and 0.004%) exhibited little ovicidal effect in laboratory conditions against green lace wing, *C. carnea*, and permitted 62.5% to 82.5% egg hatching. In contact toxicity experiments, lesser

than 10% mortality was observed, irrespective of acetamiprid concentration. In feeding toxicity test 22.5% mortality was observed at 0.004% acetamiprid and 7.5% mortality at 0.001% acetamiprid (Uthamasamy *et al.*, 2003). At 0.002 % concentration, acetamiprid was safer to the aphid predators *viz*., *C.* carnea and *M. sexmaculata* and *C. transversalis* than organophosphate insecticides like chlorpyriphos (0.05%), profenofos (0.05%) and triazophos(0.05%) on cowpea (Varghese, 2003).

The effect of the neonicotinoid insecticide, acetamiprid were tested on immature stages and adults of the parasitoid *Aphtyis melinus* DeBach (Hymenoptera: Aphelinidae), a key natural enemy of California red scale, *Aonidiella aurantii* (Maskell) (Hemiptera: Diaspididae), in California. Fourty eight hours of exposure of adults to acetamiprid residues resulted in high levels of wasp mortality. The authors conclude that treatments of acetamiprid should be avoided in situations where biological control by this parasitoid is important. (Rill *et al.*, 2008).

Nandihalli (2009) reported that, indoxacarb 14.5SC, acetamiprid 20SP and a combination product of indoxacarb 14.5 SC + acetamiprid 7.7 SC were safe to the coccinellid population found in chilli ecosystem.

### Thiamethoxam

Thiamethoxam @ 50 g a.i.  $ha^{-1}$  resulted in 25.8 per cent mortality of *Oxyopes javanus* Thorell seven days after treatment in cotton. (Mathirajan, 2001).

Al-Deeb et al. (2001) reported that thiamethoxam @ 0.002% caused significantly less mortality to *Orius insidiosus* Say adults than imidacloprid. However, thiamethoxam and imidacloprid were more toxic to green mirid bug and brown mirid bug under green house condition (Lekshmi *et al.*, 2001). Vadodaria *et al.* (2001) reported that seed treatment with thiamethoxam

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(Cruiser) 70 WS at 2.8 and 4.3 g kg<sup>-1</sup> didn't affect the natural enemy population in cotton.

Mathirajan and Regupathy (2002) revealed that thiamethoxam 25 WG (0.2 ml  $1^{-1}$ ) and oxy demeton methyl (1ml  $1^{-1}$ ) affect adult emergence, adult longevity and fecundity of *C. carnea*. Toxicity of thiamethoxam was low to moderate to aphid parasitoid, *Aphelinus gossypii* Timberlake and was high to *Delphastus pusillus* Leconte, a white fly predator (Torres *et al*., 2003).

#### 2.3.2.2 Microbial Fermentation products

#### Spinosad

spinosad 48SC at 75 and 100g ai ha<sup>-1</sup> caused 25 and 26.2 per cent mortality after 72 hr of treatment whereas chlorpyriphos20EC, quinalphos 25 EC and endosulfan 35EC) gave 100 per cent mortality after 48 hr of treatment. Spinosad was thus found to be safer to *Apis florae*, a pollinator of many agricultural crops (Mathirajan, 2002).

Spinosad was found toxic to *C. carneae* adults when applied topically and fed the product in drinking water. (Medina *et al.*, 2003).

Spinosad (0.001, 0.002, 0.003 and 0.004%) exhibited little ovicidal effect in laboratory conditions against green lace wing, *C. carnea* permitting 65.0 to 82.5% egg hatch. In contact toxicity experiments mortality ranged from 7.5 to 27.5%, depending on spinosad concentration. Spinosad exhibited less than 10 % mortality in feeding toxicity tests (Uthamasamy *et al.*, 2003). Different pesticides were sprayed on okra to study the safety against lady bird beetle, green lace wing and spider during the year 2001-2002 and 2002-2003. Spinosad 45 EC @ 75 g.a.i./ha was found most safer insecticides to predators on okra (Shinde *et al.*, 2007). In a field trial to assess the bio efficacy of newer insecticides against okra shoot and fruit borer, spinosad recorded higher population of coccinellids and *C. carnea* larvae and was found to be a safe insecticide. (Hirekurbar and Ambekar, 2008)

#### 2.3.2.3 Keto –enase group

#### Spiromesifen

In a field trial conducted to evaluate the efficacy of spiromesifen (Oberon-240SC) against the red spider mite of brinjal, it is reported that the chemical was found very safe to natural enemies (*Stethorus* sp, *Chrysoperla* sp, *Amblyseius* sp) (Sekh *et al.*, 2007).

Studies to compare the field withered toxicity/ safety of pesticides against honey bee *Apis cerana cerana* Fab and *Apis mellifera* L on mustard revealed the following order of safety after 120 hours of spraying ; imidacloprid seed treatment> spiromesifen > *Bacillus thuringensis* var. kurstaki > endosulfan> imidacloprid foliar application> cypermethrin+ *Bacillus thuringensis* var. kurstaki > lambda cyhalothrin + azadirachtin> cypermethrin> l-cyhalothrin.(Choudhary *et al.*, 2009). Spiromesifen 240 SC did not cause adverse effect on common predators viz, coccinellid beetles and green lace wings. (Ameta *et al.*, 2010).

### 2.3.2.4 Propargite

According to Kilany et al., 1996 feeding of Omite 57 EC treated nymphs of *Tetranychus urticae* had least adverse effects on the growth and fecundity of predatory mite *Amblyseius swirski* Athias – Henriot.

# 2.4 PERSISTENCE AND DEGRADATION OF INSECTICIDE RESIDUES ON CHILLI FRUITS.

Residues in chillies have been reported by various workers in India (Awasthi *et al.*, 2001, Dhotre *et al.*, 2001, Joia *et al.*, 2001). Of the sixteen consignments of agricultural produce exported from India (2000 to 2004) which was rejected by the importing countries (UK, Italy, Netherlands, Spain, Greece and Germany) eleven were Chillies/Chilli powder/ crushed chillies. Aflatoxins, Sudan red and residues of insecticides were the chief contaminants detected in the exported chilli. Among the insecticides detected ethion, triazophos, chlorpyriphos, cypermethrin and dicofol exceeded maximum residue limit (MRL) (Sreenivasa Rao, 2005).

Out of eight chilli samples collected from six agro climatic zones of Assam, three of them were contaminated with monocrotophos, but none of the samples monitered exceeds the MRL (Deka *et al.*, 2005). Six samples of green chilli were found contaminated with ethion (0.015-5.064 mg kg<sup>-1</sup>) and two samples contained residues above MRL in farm gate vegetable samples collected from farmers field in three districts of Bihar during 2003-2004 (Singh *et al.*, 2006).

### 2.4.1 Conventional insecticides on chilli fruits

#### 2.4.1.1 Organochlorines

#### Dicofol

The dissipation of dicofol 5 WP residues in chilli followed first order reaction kinetics with the degradation rate constant of 0.2792 and 0.2334 day for the respective treatments, 75 and 150 g a.i. ha<sup>-1</sup>. The actual half –life values were found to be 2.48 and 2.97 days, whereas the modified half life values were 2.31 and 2.25 days in recommended and double dose respectively (Pal *et al* ., 2005). Dicofol 18.5 EC applied @ 450 g a.i. ha<sup>-1</sup> to chillies was having an initial deposit 2.49 mg kg<sup>-1</sup>. The waiting period (T tol) for dicofol in chillies was worked out as 4.1 days respectively. (Reddy *et al.*, 2007b).

## 2.4.1.2 Organophosphates

# Dimethoate

Dimethoate 30 EC @ 300 g a.i. ha<sup>-1</sup> to chillies was having an initial deposit 0.33mg kg<sup>-1</sup>. The waiting period (T toi) for dimethoate in chillies was worked out as 1 day (Reddy *et al.*, 2007b).

Ethion

The four concentrations of ethion applied @ 500, 750, 1000, and 2000 g a.i. ha<sup>-1</sup> on chillies have the initial deposits of 5.30, 9.46, 14.91 and 19.18 mg kg<sup>-1</sup> respectively. The results of analysis reveal that the dissipation of ethion from green chillies is very fast and the residues were not detectable at 3, 6, 10, and 15 days from respective dosages. A waiting period of 3, 6, 10 and 15 can be suggested for dosage levels of 500, 750, 1000 and 2000 g a.i. ha<sup>-1</sup> respectively (Joia *et al.*, 2001).

Dissipation of ethion (0.1%) and chlorpyriphos (0.05%) on chillies was studied by spraying the crop four times at fort nightly intervals starting from 45 days after transplanting. An initial deposit of 1.84 mg kg<sup>-1</sup> of ethion and 0.67 mg kg<sup>-1</sup> of chlorpyriphos dissipated to 0.17 mg kg<sup>-1</sup> (90.8%) and 0.07 mg kg<sup>-1</sup> (89.6%) by 30 days after fourth spray respectively. Half life (RL50) values of 9.4 and 9.9 days and the waiting period (T<sub>tol</sub>) of 7.16 and 21.2 days were calculated for ethion and chlorpyriphos, respectively (Mahalingappa *et al.*, 2006).

#### 2.4.1.3 Synthetic pyrethroids

#### Lambda Cyhalothrin

Three field trials were conducted during January-March (1997) June – Sep (1997) and January-March (1998) at Coimbatore to study dissipation pattern of lambda cyhalothrin in/on chillies at 7.5, 15 and 30 ga.i.ha<sup>-1</sup>. The lower dose reached below detectable level (BDL) on the 7 day after spraying whereas at medium and higher doses the residue reached BDL on 10 days after spraying. The half-lives of residues ranged between 0.77-0.93, 0.81-0.97 and 0.82-0.99 days for corresponding doses of 7.5, 15 and 30g a.i.ha<sup>-1</sup> (Mathirajan *et al.*,2000).

# 2.4.2 Conventional insecticides in other fruits and plant parts

# 2.4.2.1 Organochlorine

#### Dicofol

Trials conducted to study the persistence of dicofol (Kelthane 18.5EC) at the rate of 0.05% and 0.1% in/on papaya resulted in an initial residues of 3.14 and 5.97ppm and the residues dissipated at the half life of 2.2 and 2.9 days respectively (Ahuja *et al.*, 2005).

# 2.4.2.2 Organophosphates

# Ethion

Application of ethion @ 0.1% on the petals of rose flower reported an initial deposit of 2.32 mg/ kg and reported a waiting period of five to six days ( Akashe *et al.*, 2002). Residues of ethion were estimated in cucumber following three applications of the insecticide at 375 and 750 g a.i. ha<sup>-1</sup>. The average initial deposit of ethion on cucumber fruits was 2.40 and 4.97 mg kg<sup>-1</sup> at single and double dosages, respectively. The residues dissipated below maximum residue limit (MRL) of 0.5 mg kg<sup>-1</sup> in 7 days at recommended dose. Half life for degradation of ethion on cucumber was observed to be 2.92 days at recommended dosage. A waiting period of 7 days is suggested for safe consumption of cucumber (Singh *et al.*, 2007). The application of ethion @ 375 and 750 g a.i. ha<sup>-1</sup> resulted in initial deposits of 2.01 and 4.93 mg/kg on brinjal fruits. The residues were below detectable level at 4 and 7 days of application at minimum effective and double effective doses respectively. A waiting period of 4 days was suggested for safe consumption of brinjal fruits (Jyot et al., 2005).

### Dimethoate

An experiment was conducted to study the persistence of dimethoate (Rogor 30 EC) at the rate of 0.05% and 0.1% respectively in/on papaya. Initial residues of dimethoate were 2.96 and 4.23 ppm and the residues persisted for 25 days. The residues dissipated at the half life ( $T_{h}$ ) of 3.7 and 5.04 days and suggested a pre harvest interval (PHI) of 3 and 5 days (Ahuja *et al.*, 2005).

# 2.4.3 New generation insecticides in chilli

# 2.4.3.1 Neonicotinoids

#### Imidacloprid

In green chillies, the initial residues of imidacloprid were 0.38 and 0.56 ppm in a spray treatment of 100 and 150 ml ha<sup>-1</sup> respectively and these residues reached below detectable limit (BDL) of 0.05 ppm in 4.19 to 5.48 days. In case of higher rate of 300ml/ha, the residues dissipated in 8.16 days and the residue half life was in the range of 1.47 to 1.78 days (Kharbade *et al.*, 2003).

# Acetamiprid

Acetamiprid 20% SP and acetamiprid 20 % SL applied in chilli @ 20 g a.i. ha<sup>-1</sup> and 40 g a.i. ha<sup>-1</sup> recorded initial deposits of 0.0207, 0.0405, 0.0244 and 0.1039  $\mu$ g g<sup>-1</sup> respectively. The half life values of acetamiprid in chilli were in the range of 2.24-4.84 days and a waiting period of 1 day is suggested for safe consumption of chilli fruits (Sanyal *et al.*, 2008).

# 2.4.3.2 Keto - enase group

#### Spiromesifen

When sprayed at 96 g a.i.  $ha^{-1}$ , the initial residues of spiromesifen (Oberon 240 SC) in chilli were in the range of 0.51- 0.56 mg kg<sup>-1</sup>. Up to 95% residues were degraded within 10 days after application. The DT50 values of spiromesifen in chilies were calculated as 2-2.5 days. (Sharma *et al.*, 2007).

# 2.4.3.4 Pyroles

# Fipronil

Dissipation of fipronil @ 0.01% and profenofos @ 0.1% on chillies were studied by spraying four times at 15 days interval starting from 45 days after transplanting. The initial deposits of fipronil and profenofos after last spray were 0.20 and 0.36 mg kg<sup>-1</sup> which dissipated to 0.01 and 0.02 mg kg<sup>-1</sup> by 30 days amounting to the loss of 94.0 and 92.4 % respectively. Half life values for fipronil and profenofos were 16.8 and 41.0 days respectively. Waiting periods of 5 and 19 days have been suggested (Reddy *et al.*, 2007b).

# 2.4.5 New generation insecticides in/ on other fruits or plant parts

# 2.4.5.1 Neonicotenoids

#### Imidacloprid

In okra, when imidacloprid was given as seed treatment @ 3, 5.4, 10.8 and 21.6 g a.i. kg<sup>-1</sup> of seeds, the residues were found to be 0.08, 0.10, 0.14 and 0.24 mg kg<sup>-1</sup> respectively after 55 days of sowing and the residues became non detectable after 60 days of sowing (Dikshit *et al.*, 2000).

When applied at the rate of 20 and 40 g a.i. ha<sup>-1</sup>, the initial deposit of imidacloprid in tomato fruits was 1.35 and 2.40 mg kg<sup>-1</sup>, respectively. The imidacloprid residues reduced progressively with time and on the seventh day the concentration was 0.08 and 0.018 mg kg<sup>-1</sup> from respective treatments and become non detectable on the tenth day from the normal dose (20 g a.i.ha<sup>-1</sup>) and the safe waiting period was seven days after treatment (Dikshit and Pachauri, 2000).

The residues of imidacloprid, Confidor 200SL at 0.3 and 0.6 ml  $L^{-1}$  dissipated exponentially with time following foliar application with half-life of 2-4 days in two consecutive seasons. The residues became non- detectable at 10 days after treatment at lower concentration and 15 days after treatment at higher concentration (Indumathi *et al.*, 2001). Residues of imidacloprid in

chilli fruits were estimated by the HPLC at the sensitivity of 0.05ppm. Initial residues on green chillies after third spray at the rate of 100 and 150ml were 0.38 to 0.58ppm, which dissipated to below detectable limit of 0.05ppm in 4.19 to 5.48 days. The residue half life was in the range of 1.47 to 1.78 days (Kharbade *et al.*, 2003).

Experiments were conducted to study the persistence of imidacloprid, acetamiprid and thiamethoxam in gram following seed dressing (3 and 6 g a.i. kg<sup>-1</sup> seed) and foliar application (imidacloprid @ 20 and 40 g a.i.ha<sup>-1</sup> and acetamiprid and thiamethoxam @ 25 and 50 g a.i.ha<sup>-1</sup>). Following seed dressing, the residues in green plant persisted beyond 30 days after sowing. However, no residue was detected in green plants after 45 days. Following foliar spray, imidacloprid persisted beyond 3 days but no residues were detected on fifth day. Similarly, the residues of thiamethoxam and acetamiprid persisted beyond 5 days but no residues was detected on the tenth day for high dose of thiamethoxam (Gupta *et al.*, 2005).

Imidacloprid (Confidor 200 SL) was drenched in apple tree basin @890 g a.i. ha<sup>-1</sup> (20 ml / 4L and @ 1780 g a.i.ha<sup>-1</sup> at fruit development stage in three different locations of Himachal Pradesh. Fruit samples were collected at 10, 20, 30 and 40 days whereas soil samples were collected at 40 days after drenching. Imidacloprid residues ranging from 2.34-5.49 and 3.62-6.08 mg kg<sup>-1</sup> were detected in fruit samples collected after 10 and 20 days of drenching respectively, whereas residues were non detectable on 30 and 40 days (Dubey *et al.*, 2006).

Following application of Confidor 350 SC @ 26.25 and 52.50 g a.i.ha<sup>-1</sup> and Confidor 70 WG @ 24.5 and 49.0 g a.i. ha<sup>-1</sup> at fortnightly interval after 45 days of transplanting paddy, the residues of imidacloprid was below limit of determination of 0.01, 0.05, 0.05 and 0.01 mg kg<sup>-1</sup>, in rice grain, straw, husk and soil respectively on both dosages at harvest time (Kang *et al.*, 2007). Imidacloprid was sprayed at two dose levels, 125 (recommended dose) and

250 ml ha<sup>-1</sup>(double of the recommended dose). Half lives of imidacloprid in green shoots were in the range of 1.14-1.23 and 1.03-1.09 days, and 1.14-1.25, 1.04-1.07 days in made tea, for the dry and wet season, respectively. The per cent transfer of imidacloprid residue from made tea to infusion was in the range of 29.2- 42.0% during the dry and wet seasons; however, 38.2% and 57.9% of the residues remained stuck to the spent leaves during the dry and wet seasons, respectively. On the basis of transfer from made tea to hot water infusion, a waiting period of 7 days after pesticide application at a recommended dose for tea plucking is suggested (Gupta *et al.*, 2007). Residues of imidacloprid (seed treatment @ 21 g a.i. ha<sup>-1</sup>) were neither found in nectar nor in pollen samples of *Brassica juncea* (L) at the time of sampling i.e. 50% flowering (Choudary and Sharma, 2008).

#### Acetamiprid

Studies on the persistence of acetamiprid in green gram following seed dressing (3 and 6 g a.i. kg<sup>-1</sup> seed) and foliar application of acetamiprid @ 25 and 50 g a.i.ha<sup>-1</sup> revealed that following seed dressing, the residues in green gram persisted beyond 30 days after sowing, while no residue was detected in green plants after 45 days. Following foliar spray, the acetamiprid residues persisted upto 5<sup>th</sup> day of spraying and the residues were below detectable level on 10 days after spraying (Gupta *et al.*, 2005). Acetamiprid (Pride 20 SP) applied @ 75 g a.i.ha<sup>-1</sup> on okra fruits recorded an initial deposit of  $0.335\mu g g^{-1}$  and more than 50% dissipation was observed on third day after application. Residues were below detectable limit on seventh day after application and an half-lfe of 2.3days was calculated on okra fruits (Singh and Kulshrestha, 2005).

The harvest time residues of acetamiprid 20 SP applied @10, 20, 40 and 80 g a.i.ha<sup>-1</sup> on cotton variety RCH2 were found to be below detectable level of 0.5 ng in lint, soil, seed and oil (Kanna et al., 2007).

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# Thiamethoxam

Experiments were conducted to study the persistence of thiamethoxam in gram following seed dressing (3 and 6 g a.i. kg<sup>-1</sup> seed) and foliar application of thiamethoxam @ 25 and 50 g a.i.ha<sup>-1</sup>. Following seed dressing, the residues in green plant persisted beyond 30 days after sowing while no residues were detected in green plants after 45 days. Following foliar spray the residues of thiamethoxam persisted upto 5 days for lower dose, but the residues were detected on the  $10^{th}$  day for higher dose of thiamethoxam (Gupta *et al.*, 2005).

### 2.4.5.2 Keto-enase group

#### Spiromesifen .

Initial deposit of spiromesifen when applied @ 96 g a.i ha<sup>-1</sup> on apple was in the range of 0.88- 0.99 mg kg<sup>-1</sup> at different locations. Half- life of the insecticide on apple evaluated at different locations was found to be in the range of 5-6 days (Sharma *et al.*, 2005).

Half life (T<sub>1/2</sub>) of spiromesifen applied on tea @96 g a.i. ha<sup>-1</sup>was found to be 5.0-5.8 days. Ninety nine percentage degradation was found to occur within 33-57 days after application. (Sharma *et al.*, 2006a).

The average half life of spiromesifen residues in nectar and pollen of mustard (*Brassica juncea* (L) Czern.) were 19.99 and 9.69 h respectively. A waiting period of 5 days should be observed on crops sprayed during blooms to avoid any accidental hazards to honey bees (Choudary and Sharma, 2008)

#### 2.4.5.3 Propargite

In soil, half- life of propargite (Omite 57EC) at the recommended and double dosages of 0.028 and 0.06 % ranged from 43 to 45 days. The half life calculated in apple fruit at recommended dose was 2.61 days while in tea it was 1.66 days. The percent transfer of propargite residue from manufactured

tea to infusion was 23.6 to 40.0. However, 35.71 to 53.20 % of residues remained stuck to the spent leaves (Kumar et al., 2005a).

Residues of propargite estimated in brinjal fruits following application of Omite 57EC @ 570 and 1140 ga.i.ha<sup>-1</sup> indicated that the average initial deposits were 0.51 and 0.92 mg kg<sup>-1</sup> respectively which were below the maximum residue limit of 2 mg kg<sup>-1</sup>. The half life values ( $T_{1/2}$ ) of propargite were worked out as 3.07 and 3.54 days respectively and a waiting period of one day has been suggested for safe consumption of fruits (Kang *et al.*, 2009).

# 2.5 EXTENT OF REMOVAL OF INSECTICIDE RESIDUES BY HOUSE HOLD PRACTICES.

## 2.5.1 Washing

Washing is the preliminary cleaning step in both household and commercial preparations. Loosely held residues of several pesticides are removed with a reasonable efficiency by varied types of washing processes (Street, 1969). Unwashed samples of green beans contained an average of 1.49 ppm of EDBC (Ethylene bis dithiocarabamate) and very low levels of its metabolite ETU (ethylene thiourea). Washing of beans in cold water for 2minutes removed 45% of EDBC but didn't affect the levels of ETU. The wash with alkaline hypochlorite followed by dipping in dilute sodium sulfite left no detectable residues of EDBC or ETU on the beans (Marshall, 1982).

Bitter gourds treated with endosulfan sprays received initial deposits of 18.97 and 26.01 ppm which were respectively removed to extent of 59.05% and 42.66 % by 30 s of washing (Nath and Agnihotri, 1984). Of dithane-M-45 applied to egg plant (179 ppm), 47.5 and 22.3 % remained on unwashed and washed fruits, respectively 15 days after harvest (Kumar and Agarwal, 1991). Chlorpyriphos and its breakdown products 3, 5, 6-trichloro-2-prydinol were recovered from fortified rice grains in the levels of 456 and 3.4 ppb, respectively. Washing rice grains with water removed approximately 60% of the chlorpyriphos residues (Lee *et al.*, 1991).

Washing of mango fruits for 10 minutes reduced residues to 66-68 % for dimethoate and fenthion as against 21-27% for fenvalverate and cypermethrin (Awasthi, 1993). From an initial level of 19 ppm in rice almost all the permethrin in rice was removed by washing with water (Fukuhara *et al.*, 1994).

Holland *et al.*, (1994) reported that washing can reduce 47 % of parathion residues in cow pea and cauliflower. Washing also reduced 37% parathion residues in spinach.

Washing of soybeans twice with water reduced the pesticides by 80-90% of the initial levels of 5.01ppm dichlorvos, 7.9 ppm malathion, 11.2 ppm chlorpyriphos and 2.87 ppm captan. These results suggest that sprayed pesticide remain as micro particles on the surface of soybeans and are easily removed by mechanical stirring in water (Miyahara and Saito, 1994).

The concentration of iprodione in peaches was 1.23 ppm which in washed fruits reduced to 0.61 ppm (Lentza-Rizos, 1995). Washing of Golden delicious apples brought about reduction of 30-50% in phosalone residues. (Mergnat *et al.*, 1995). Washing coupled with gentle rubbing by hand under tap water for 1 minute dislodge pesticide residue on brinjal (Barroah and Yein, 1996).

In a trial to study the influence of age of residues and different types of wash on residue removal, it is revealed that residues get reduced from 0 day to  $5^{\text{th}}$  day and tamarind solution 2% proved best in reducing residues at 0 and  $5^{\text{th}}$  day (Gopichand *et al.*, 1999). Almost 53% of azinphos-methyl residue was removed from fruit with the water wash. Apples dipped in ozonated water (0.25 ppm) had reduced residue levels by about 75%. Chlorine wash at 50 and

500 ppm removed about 76 per cent and 83 per cent of pesticide residue, respectively (Ong *et al.*, 1996).

Kumar, (1997) reported that dipping in 2% salt water for 1 hour removed more than 90% residues of monocrotophos and phosphamidon in bitter gourd. The same author also reported the effectiveness of tamarind water, lime water and vinegar in reducing the pesticide residues.

Tomatoes contaminated at level of  $1\mu g g^{-1}$  up on washing with different levels of acetic acid solution gave 51.3, 47.0, 33.7, 91.5, 86.0 and 93.7 % loss in HCB, lindane, p,p-DDT, dimethoate, profenofos and primiphos-methyl respectively. Sodium chloride (10 %) washing came next in importance to washing by acetic acid solutions giving 42.9, 46.1, 27.2, 90.8, 82.4 and 91.4 % loss in the same pesticides respectively. On the other hand washing by tap water proved the least effective, showing 9.62, 15.3, 17, 18.8, 22.7 and 16.2 % loss of HCB, lindane, pp-DDT, dimethoate, profenofos, and primiphos-methyl, respectively (Abou –Arab, 1999).

Senapathi *et al.*, (1999) reported that 1% alcohol was very effective in reducing residues of quinalphos and endosulfan in cauliflower followed by 0.05% NaHCO3 and 0.2 % NaCl. The total amount of residue on unwashed fruit was determined to be 0.67ppm. Studies with tomatoes fortified with (14C) ETU (0.006 ppm) showed that 70% of the radioactivity was lost during washing of the fruits in water (Knio *et al.*, 2000).

Twelve pesticides, which include the fungicides captan, chlorothalonil, iprodione, vinclozolin, the insecticides endosulfan, permethrin, methoxychlor malathion, diazinon, chlorpyrifos, bifenthrin and DDE (a soil metabolite of DDT) were selected to initiate a research programme with the objective of examining the effect of rinsing of produce with tap water to reduce pesticide residue on it. Statistical analysis of the data showed that rinsing removed residues of nine of the twelve pesticides studied. Residues of vinclozolin, bifenthrin and chlorpyrifos were not reduced. The rinsability of the pesticides is not correlated with its water solubility (Krol *et al.*, 2000).

The effect of washing procedures on pesticide residues in potato was studied. Potatoes were contaminated with lindane, aldrin and heptachlor epoxide at levels of 13.8, 2.5 and 14.5 ppm, respectively. Washing with 5 and 10% radish solution completely removed aldrin, lindane and heptachlor epoxide. Similar results were obtained by washing with 10 % ascorbic acid and 10% citric acid solutions but the reduction in aldrin was 85-90%. Alkaline solution of 10% sodium carbonate led to 92, 88, and 95 % removal of residues, while neutral solution of 10% sodium chloride removed only 42, 76 and 86 % of the residues of the insecticides respectively. Tap water was least effective in reducing pesticide residues and removed them by only 10-12% (Zohair, 2001).

Dipping of grape berries in 2% salt solution for 10 minutes followed by washing with water proved to be an effective decontamination procedure, facilitating removal of 67.52 and 6.5 per cent of residues of chlorpyrifos and 51.77 and 50 per cent quinalphos residues correspondingly at 1 and 5 day after spraying (Reddy and Rao, 2002).

Washing of chickpea grains reduced the deltamethrin residues by 15.69% from an initial level of 0.051% (Lal and Dikshit, 2001). Washing of okra fruits with tap water removed cypermethrin residues to the extent of 41.2-48.3% in 0 day samples and 37.1 to 46.0 in 5<sup>th</sup> day samples. In the case of fluvalinate, the extent of removal was 38.0-44.2% in 0 day and 32.4-41.8% in fifth day samples (Singh *et al.*, 2004b).

The initial diazinon residue level (0.822 ppm) on cucumber was decreased by 22.3% by washing for 15 seconds by rubbing under running water (Cengiz *et al.*, 2006). The initial procymdone residue level (0.86 ppm) on tomatoes was decreased 68% by washing for 15 seconds by rubbing under running water (Cengiz *et al.*, 2007).

The effect of different household processes (washing and boiling/cooking) on reduction of residues of organochlorine (OC), synthetic pyrethroids (SP), organophosphates (OP) and carbamates were determined in three vegetables viz. brinjal, cauliflower and okra. In all the three vegetables washing reduced the residues by 20-77% and boiling by 32-100%. Maximum (77%) reduction of OP insecticides was observed in brinjal, followed by 74% in cauliflower and 50% in okra by washing. By boiling process, 100% of the residue was removed in brinjal followed by 92% in cauliflower and 75% in okra (Kumari, 2008). Captan residues in apples washed for 10-15s with continuous hand rubbing were 50% lower than in those apples that received no post harvest washing (25.5-5100 ng g<sup>-1</sup>) (Rawn *et al.*, 2008).

# 2.5.2 Washing and cooking

Washing the apples followed by cooking (including processing apple to sauce) reduced the amount of residue by 98%. The total amount of residue on unwashed fruit was determined to be 0.67 ppm (Ong *et al.*, 1996). Kumar (1997) studied the effect of closed and open system of cooking in reducing pesticide residues and found that cooking bittergourd fruits for 25 minutes (open and closed cooking) removed residues of monocrotophos in the range of 52.2 to 54.44 per cent and phosphamidon in the range of 45.05 to 56.04 per cent.

The residues of triazophos and lindane were reduced by washing followed by cooking treatments at 0 and 5 days after spraying in okra. The percentage reduction of triazophos residues increased on fifth day (56.05%) after washing and cooking treatments than on the 0 day (39.57%) whereas the reduction of lindane residues decreased on the fifth day (28.4%) than on the day of spraying (49.23%) (Reddy *et al.*, 1999).

Washing and cooking were effective in lowering the alphamethrin (0.005%) residues in brinjal. However, reduction of residues was more due to cooking than simple washing (25 to 33%). In tomato, both the processes

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reduced the residues almost to the same extent (11 to 33%) (Kanta *et al.*, 2001). Washing and steaming of chickpea grains completely removed the deltamethrin residues from an initial deposit of 0.051ppm (Lal and Dikshit, 2001).

Beevi *et al.*, (2003) studied the effect of washing and cooking in removing residues of various vegetables and noted substantial reduction of pesticide residues in washed and cooked vegetables.

# 2.5.3 Washing and drying

Low level residues of abamectin on field treated tomatoes became nondetectable on the washed fruit (hot dip, pH 11) or in canned puree but were detectable on the wet pomace and residues get concentrated eight times on drying (JMPR, 1992).

The residues of iprodione in prune at harvest time was 0.68ppm which became 0.30ppm after washing with water for five minutes followed by oven drying and dehydration (Cabras *et al.*, 1998).

A laboratory experiment conducted at Agricultural research station, Durgapura, Jaipur during 2006 to find out the effect of drying on the residues of dicofol (18.5 EC), ethion (50 EC) and cypermethrin (25 EC) at 0.05, 0.05 and 0.015 per cent concentration in chilli revealed that respective initial deposits of dicofol (18.5 EC), ethion (50 EC) and cypermerthrin (25 EC) in fresh chilli were 0.72, 0.40 and 0.02 mg kg<sup>-1</sup> respectively, whereas in sun dried chilli powder they were 4.03, 1.41 and 0.15 mg kg<sup>-1</sup> respectively (Pathan *et al.*, 2009).

# 2.5.4 Washing, peeling and juicing

Holland *et al.*, (1994) studied the effect of juicing and vinification on pesticide residues of metalaxyl in grapes. The results showed that the residue level get reduced from must (no residue reduction) to clarified grape juice (30-50 %) and then to wine (66 %). Burchat *et al.*, (1998) studied the efficiency of

peeling in reducing residues in carrot. In peeled carrots residues were not detected and in the carrot peel residues got concentrated.

Field sprayed apples and lemons with an initial content of 1.31 ppm azinphos-methyl were used for production of juice. The apples were washed 15 min under running water and juiced. In apples on zero days the amount of pesticide found in the juice was 0.51 ppm which increased till twelfth day and decreased to 1.13 ppm on twenty-sixth day, while no insecticide residues was detected in the lemon juice produced right from zero to twenty sixth day (Athanasopoulos and Pappas, 2000).

Propineb (Antracol 70WP) residues were estimated in raw apple fruits, decontaminated fruits and processed fruit products, after application @ 2.5 and 5.0 kg a.i. ha<sup>-1</sup> on fruit bearing apple trees. Initial deposits of 5.98 and 11.0 mg kg<sup>-1</sup> in/on fruits dissipated below the MRL (3 mg kg<sup>-1</sup>) in 4.10 and 7.53 days respectively. Washing, peeling and washing + peeling of fruits were adopted to decontaminate propineb residues from apple fruits. Among the different decontamination processes washing + peeling was found to be the best technique resulted in 75.8 - 98.3% removal of residues (Kumar *et al.*, 2005b).

Vegetables pickled in rice bran for 5 minutes removed more than 85% of the residues of chlorothalonil and tetradifon. The mechanism of pesticide removal by rice bran was attributed to the uptake of pesticides into intracellular particles called spherosomes (Adachi and Okano, 2006). Captan residues in apples washed with de-ionized water for 10-15 seconds with continuous hand rubbing were 50 per cent lower than in those apples that received no post harvest washing. The reduction of residues on the apples that had been washed and peeled was greatest around 98 per cent (Rawn *et al.*, 2008).

# **MATERIALS AND METHODS**

## 3. MATERIALS AND METHODS

Laboratory and field experiments were carried out for the evaluation of few of the traditional and new generation insecticide molecules in the management of leaf curl of chilli caused by mites, thrips and aphids and their safety to non target biota. The laboratory experiments were carried out at the Department of Entomology and the Pesticide Residue Research and Analytical Laboratory under All India Network Project on Pesticide Residues, College of Agriculture Vellayani. The field trial was conducted at the instructional Farm, College of Agriculture, Vellayani.

# 3.1 LABORATORY EVALUATION OF CONVENTIONAL AND NEW GENERATION INSECTICDES AGAINST SUCKING PEST COMPLEX IN CHILLI.

Twelve insecticides were evaluated for their efficacy against the sucking pest complex in chilli. The experiments were laid out in completely randomized block design with three replications and an untreated check. The treatments are detailed in Table 1.

The three sucking pests of chilli selected for the study were

1.	Aphid	-	Aphis gossypii Glover
2.	Chilli mite	_	Polyphagotarsonemus latus (Banks)
3.	Chilli thrips	_	Scirtothrips dorsalis (Hood)

# 3.1.1 Raising of test plants

The seeds of chilli *Capsicum annum* (cv. Anugraha) were procured from the Olericulture Department, College of Horticulture, Kerala Agricultural University, Vellanikkara, Main Campus. Seeds were sown in earthen pots and 28 days old seedlings were transplanted to polypropylene Q tubs (cylindrical containers of 12cm diameter) and earthen pots (45 cm diameter) for conducting different experiments.

No	Common Name	Trade Name	Dosage	Manufacturer
1	Spinosad	Tracer 45 SC	75 g a.i.ha <sup>-1</sup>	Dow Agro Sciences India Pvt Ltd
2	Spiromesifen	Oberon 240 SC	100ga.i.ha <sup>-1</sup>	Bayer Crop Science India Ltd
3	Spirotetramat	Spirotetramat240 OD	60 g a.i.ha <sup>-1</sup>	Bayer Crop Science India Ltd
4	Indoxacarb	Avaunt 14.5 SC	60 g a.i.ha <sup>-1</sup>	E.I. DuPont India Pvt Ltd
5	Imidacloprid	Confidor 200 SL	20 g a.i.ha	Bayer Crop Science India Ltd
6	Thiamethoxam	Actara 25 WG	40 g a.i.ha '	Syngenta India Ltd
7	Flubendiamide	Fame 480 SC	100 g a.i.ha <sup>-1</sup>	Bayer Crop Science India Ltd
8	Acetamiprid	Pride 20 SP	20 g a.i.ha '	Dow Agro Sciences India Pvt Ltd.
9	Propargite	Simba 57 EC	570 g a.i.ha <sup>-1</sup>	P I Industries Ltd
10	Dimethoate	Rogor 30 EC	300 g a.i. ha <sup>-1</sup>	Rallis India Ltd
11	Ethion*	Fosmite 50 EC	375 g a.i.ha <sup>-1</sup>	P I Industries Ltd
12	Oxy demeton methyl*	Metasystox 25 EC	500 g a.i.ha <sup>-1</sup>	United Phosphorus Ltd

Table 2. List of the insecticides evaluated for their efficacy against thrips, mites and aphids.

\* The insecticidal checks included in the field experiment.

# 3.1.2 Evaluation against aphids

Twigs of chilli infested with *A. gossypii* were collected from unsprayed fields of the instructional farm, Vellayani and aphids were allowed to infest chilli plants raised in earthen pots as described in 3.1.1. Two months old chilli plants raised in poly propylene Q- tubs (Plate 3a) of 12 cm diameter were

protected using cylindrical polyester cages having cloth lined ventilations to prevent the entry of other insects. Adult aphids were collected from potted chilli plants maintained in the laboratory. Twenty five adult aphids were released into these caged plants (Plate 3b) and different insecticidal treatments were given using hand sprayers. Observations were recorded upto 4 days after spraying (DAS). The percentage mortality was corrected using Abbot's formula (Abbot, 1925).

# 3.1.3 Evaluation against mites and thrips using leaf disc method

Young leaves collected from 3-4 months old chilli plants were dipped in pesticidal spray solution and air dried. Leaf discs of 2cm diameter were prepared from these leaves using sharp edged metallic punch. Cotton pad was placed in petridish (9cm diameter) and moistened with water to saturated level. These treated discs were transferred to each cotton pad using a forceps and placed equidistantly in an upside down position (Plate 4).

Adult mite and thrips were collected from potted chilli plants maintained in the laboratory exposed to natural infestation. The chilli mites were individually collected from infested curled leaves using a soft and fine bristled brush moistened with water. These mites were released into treated leaf disc. This procedure was done with the help of a stereo binocular microscope. Leaf discs were inspected at 24, 48 and 72 hrs. The percentage mortality was corrected using Abbots formula (Abbot, 1925). For thrips, the same methodology was adopted with the only exception that 2<sup>nd</sup> instar nymphs were used.

# 3.1.4 Evaluation of chilli mites in potted plants

Potted plants infested by chilli mite, *P. latus* were selected for evaluation. These plants were given insecticidal sprays using hand sprayer. The mite count, both adult and nymph were taken one day before spraying (pre count) and 1, 3 and 7 days after spraying. Five leaves on the top canopy



Plate 3a. Chilli plants raised in Q tubs for evaluation against *Aphis gossypii* 



Plate 3b. Caged chilli plants for evaluation against *Aphis gossypii* 

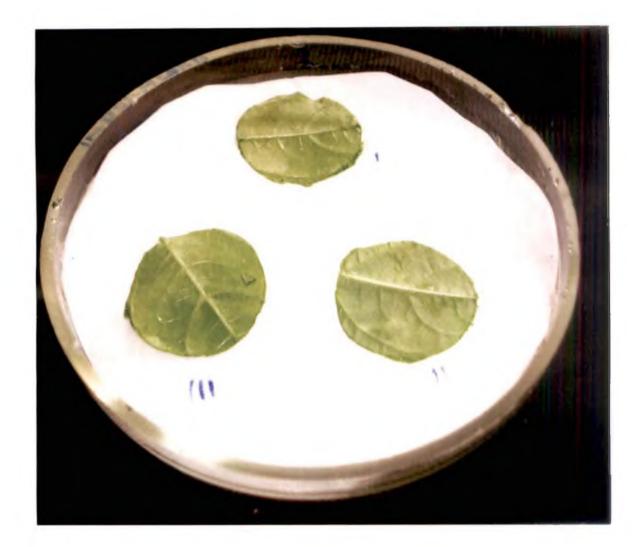


Plate 4. Petri dish containing insecticide treated leaf discs of chilli exposed to *Polyphagotarsonemus latus* and *Scirtothrips dorsalis* 

of each plant were selected at random for counting mites. The mite count was taken with the help of a stereo binocular microscope.

# 3.1.5 Evaluation of chilli thrips in potted plants

Potted plants infested by chilli thrips, *S. dorsalis* were selected for evaluation. These plants were given insecticidal sprays using hand sprayer. The thrips count, were taken one day before spraying (pre count) and 1, 3 and 7 days after spraying. Five leaves on the top canopy of each plant were selected at random for counting thrips. The thrips count was taken with the help of a stereo binocular microscope.

# 3.1.6 Leaf curl index

Leaf curl index was worked out to assess the extent of damage caused by chilli mites and thrips. Leaf damage was scored visually following the standard scoring procedure mentioned below.

Table 3. Leaf Curl Index in Chilli

Score	Symptom					
0	No symptom					
1	1-25% leaves / plant show curling					
2	26-50% leaves / plant show curling-moderately damaged					
3	51-75% leaves / plant show curling, heavily damaged, malformation of growing points and reduction in plant height					
4	>75% leaves / plant show curling severe and complete destruction of growing points, drastic reduction in plant height, defoliation and severe malformation					

# 3.2 EVALUATION OF SAFETY OF INSECTICIDES TO NATURAL ENEMIES

The insecticide molecules evaluated against chilli mites and thrips in the laboratory were further evaluated for their safety to major predator of thrips, mite and aphids.

#### 3.2.1 Safety evaluation against predatory mite

The predatory mites used for the evaluation were collected from infested chilli plants maintained in the laboratory (Plate 5a). Safety evaluation against predatory mites was done with the same methodology described in 3.1.3.

#### 3.2.2 Laboratory rearing of predators

#### 3.2.2.1 Coccinellid grubs

Aphid infested twigs/branches of chilli plants were excised from culture plants maintained in the laboratory as described in 3.1.2. The cut end of the twigs was covered with moistened cotton and inserted into small glass vials containing water. The twigs along with the vials were placed in cylindrical  $20 \times 15$  cm glass troughs covered with muslin cloth. The beetles were introduced in to the troughs containing their respective prey for mating and egg laying. The neonate larvae of the coccinellids (Plate 5b) were transferred to similar glass troughs containing their respective aphid prey. The troughs were cleaned and replenished with sufficient aphid prey daily. The newly moulted third instar larvae from the culture were used for the various studies.

# 3.2.2.2 Hemerobid larvae

Twigs of chilli plants with aphid colonies harbouring hemerobid larvae were collected from unsprayed chilli fields. The larvae were separated from the colonies and introduced individually into 5 cm glass vials with aphid prey



А



В



Plate 5. Natural enemies of sucking pests, (A) *Amblyseius* spp (B) Coccinellid grub (C) Hemerobid larvae used for laboratory evaluation till pupation. The vials were replenished with aphid prey regularly. The pupae were kept together in separate container and the freshly emerged adults were transferred to clean troughs provided with honey solution for mating and oviposition. The larvae were maintained in the troughs with sufficient prey till the fourth instar. From the fourth instar onwards, they were reared individually in separate vials as detailed under item 3.2.2.2 to avoid cannibalism. The third instar larvae (Plate 5c) were used for the study.

### 3.2.3 Safety evaluation against coccinellid grubs and hemerobid larvae

The laboratory reared third instar grub of coccinellid beetles and third instar larvae of hemerobids were used for the study. Five young ones of these insects were released separately to insecticide treated, air dried chilli leaves (with moistened cotton swab at petiole end) in a petri dish (9 cm diameter). The young ones of the natural enemies were provided with aphid prey daily to avoid starvation. Observations on the mortality of the predators were taken at 24, 48 and 72 hrs after exposure to insecticides. The mortality was corrected using Abbot's formula (Abbot, 1925).

# 3.3 FIELD EVALUATION OF CONVENTIONAL AND NEW GENERATION INSECTICIDES AGAINST SUCKING PEST COMPLEX IN CHILLI

From the ten insecticides evaluated in the laboratory against mite, thrips and aphids, infesting chilli, six insecticides (five new generation insecticides along with one conventional insecticide) were selected for the evaluation in the field. Two additional insecticides were also included in the treatment (Table 1) to make altogether eight treatments. The experiments were laid out in randomized block design with three replication and an untreated check.

# 3.3.1 Field Experiment

The seeds of chilli var Anugraha procured from the Olericulture Department, College of Horticulture, Kerala Agriculture University, Vellanikara, Main Campus were sown in two plots of size 2×2 sq m. One month old seedlings were transplanted during the last week of February 2009 in plot size of 2×2 sq m with a spacing of 45×45cm. Each plot had a density of 20 plants with one plant per hill. All the management practices except the plant protection against the sucking pests in chilli were followed as per the recommended package of practices of Kerala Agricultural University (KAU,2007).

Following insecticides were selected for the management of mites and thrips in field study

- Spiromesifen (Oberon 240 SC)
- Imidacloprid (Confidor 200 SL)
- Thiamethoxam (Actara 25 WG)
- Acetamprid (Pride 20 SP)
- Propargite (Simbaa 57 EC)
- Ethion (Fosmite 50 EC)
- Dimethoate (Rogor 30 EC)
- Oxy demeton methyl (Metasystox 25 EC)

The insecticidal sprays were given at 30, 60 and 90 DAT. Spraying was done using a pneumatic knapsack sprayer.

# 3.3.2 Population density of mite and thrips

Mite and thrips count were taken 1, 3,5, 7 and 14 day after spraying. For counting the population of mites and thrips numbers six plants were selected at random in each plot and tagged. Six leaves were collected at random from the top canopy of each selected plant and brought to the laboratory in zip lock bags and observed under stereo binocular microscope.

# 3.3.3 Leaf Curl Index

Ten plants were selected randomly in each plot and scored for leaf curling visually following the standard scoring procedure mentioned in table 3, at 50, 70 and 100 DAT, by visual observations.

# 3.3.4 Population density of predators

The plants selected for counting the mite and thrips were observed for recording the population density of predatory mite, coccinellid beetles, spiders, neutrals and pollinators.

#### 3.3.5 Growth and yield parameters of chilli

Growth and yield parameters of chilli crop at different stages recorded were given below.

Plant height (cm) at 30, 60 and 90 days after transplanting (DAT)

Number of branches/plant at 30,60 and 90 DAT

Number of fruits/plant at 60, 90 and 120 DAT

Height of the plant from the base to the tip of the upper most branches was measured and expressed in centimeter (cm). For taking the count of branches, secondary branches were taken into account.

# 3.3.6 Fruit yield (kg / plot) and benefit cost ratio

The weight of chilli harvested were recorded and expressed as kg/plot and converted to q ha<sup>-1</sup> to calculate the benefit cost ratio. Benefit cost ratios were worked out for all the treatment applications to indicate the benefits obtained for every rupee spent. In order to obtain the benefit cost ratio the parameters, increase in yield over control, monetary returns over control and additional cost of plant protection measures were calculated. The benefit cost ratio for each treatment was obtained by dividing additional monetary returns obtained by additional cost of plant protection.

# 3.4 PERSISTENCE AND DEGRADATION KINETICS OF INSECTICIDE RESIDUES ON CHILLI FRUITS.

Persistence and degradation of seven insecticides selected based on the field evaluation were studied in the Pesticide Residue Research and Analytic Laboratory of the All India Network Project on Pesticide Residues, KAU centre at College of Agriculture Vellayani.

# 3.4.1 Field Experiment

The chilli variety Anugraha was raised in the instructional Farm, College of Agriculture, Vellayani adopting the Package of Practices Recommendations of Kerala Agricultural University (KAU, 2007). The experiment was laid out in Randomized Block Design with seven treatments in three replicates including an untreated control. One month old chilli seedlings were transplanted during the last week of November 2009 in plot of size of  $2m \times 2m$  with a spacing of  $45 \times 45$ cm. Each plot had a plant population of maximum 20 plants with one plant per hill.

Following insecticides were selected for the study on the persistence and degradation pattern of insecticides in/on chilli fruits.

L

- Spiromesifen (Oberon 240 SC)
- Acetamiprid (Pride 20 SP)
- Imidacloprid (Confidor 200 SL)
- Propargite (Simbaa 57EC)
- Ethion (Fosmite 50 EC)
- Dimethoate (Rogor 30 EC)
- Oxy demeton methyl (Metasystox 25 EC)

# 3.4.2 The glasswares, equipments and reagents which were used for conducting the various analytical procedures are listed below:-

### Laboratory Glass wares

Beaker -250 ml capacity Chromatographic column - 2.2 cm× 60 cm Class A pipette - 0.5 ml, 1ml Conical flask - 250ml, 500ml capacity Filter funnel – 75mm (dia) Graduated test tube - 5ml, 10 ml, 15 ml, 20 ml, 25ml Microsyringe -500µL Separatory funnel- 500ml, 1000ml capacity

# Laboratory Equipments

Hot air Oven Orbital Shaker (Mechanical Shaker) Vaccum flash Evoporator (Rotary Vaccum Evaporator)

# Laboratory Reagents (All distilled)

Acetone (AR Grade) Acetonitrile (AR Grade) Activated charcoal Anhydrous Sodium Sulfate (AR) Chloroform (A R Grade) Dichloromethane (AR Grade) Florisil 60-100mesh (Pesticide Residue Grade) Hexane (AR Grade) n- Hexane (HPLC) Potassium Permanganate Silica gel (60 -120 mesh) Sodium chloride

# 3.4.3 Method Validation

#### **Recovery Experiments**

Before sampling of chilli fruits from insecticide treated plots, recovery experiments were carried out to assess the efficiency of extraction and clean up procedures adopted and to standardize the procedure for pesticide residue estimation from chilli fruits. For conducting the experiment, control samples of chilli fruits were separately spiked at different levels with the certified reference material (CRM) of the seven insecticides described above. The samples were extracted and cleaned up as per the analytical procedures given below.

#### Calibration Curve

The CRM of different insecticides (> 98.00 % purity) listed above were used for making stock solution of reference analytical standard. Intermediate standards of 100 ppm were prepared from the stock solution. From this, working standards of 10 ppm and lower concentrations of 0.1, 0.2, 0.3, 0.4, 0.5, and 1 ppm were prepared. The standards of different insecticides were injected into different analytical instruments described below. A calibration curve was prepared by plotting concentration vs. peak area.

# 3.4.4 The analytical procedures for the seven insecticides were summarized below:-

The laboratory procedures for pesticide residue analysis (Sharma, 2007) of chilli fruits sample involved the basic steps of extraction, clean up, identification, quantification and confirmation.

# 3.4.4.1 Spiromesifen

#### Extraction

Chilli samples (50g) from control plots were chopped and then ground to a fine paste from which a representative sample (10 g) was taken after

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quartering. It was then spiked with analytical standard of spiromesifen at 0.01, 0.1 and 0.5 mg kg<sup>-1</sup> level. The spiked sample was further homogenized for two minutes and was extracted using a mixture of 80 ml distilled water and 100 ml of acetone. The extract was filtered through filter funnel plugged with cotton wool. The filter cake was again stirred with 100 ml acetone: water mixture (2:1, v/v) for 5 minutes and again filtered. The combined filtrate was concentrated in a rotary vacuum evaporator to half of its original volume and then transferred to a 500ml separating flask and 20 ml NaCl (10%) solution was added. The aqueous phase was partitioned twice with 100 ml hexane: ethyl acetate (1:1, v/v) followed by 75 ml of same solvent mixture. The organic phases were pooled and dried over anhydrous sodium sulphate and concentrated on rotary vacuum evaporator to about 5 ml and subjected to clean up for removal of co-extractives and impurities.

#### Clean-Up

The co-extractives were removed by adopting column chromatography using florisil. The glass column ( $60 \times 2.2$  cm) was packed compactly with activated florisil PR (10 g) sandwiched between two layers of activated anhydrous sodium sulphate (3 g). The column was pre-washed with 15ml of distilled acetone and discarded, the concentrated filtrate obtained after extraction was loaded over the column and was eluted with 100 ml of acetone at the rate of 2ml/minute. The cleaned eluate was concentrated to near dryness in a rotary vacuum evaporator and the volume was made up to 5 ml with acetone for GC-MS analysis.

#### Gas Chromatograph - Mass spectrometric Analysis

The cleaned extracts were analysed on Shimadzu GC-MS2010 QP plus equipped with a mass detector, fitted with capillary column (J &W, DB-1,MS \_ Ultra innert) of 30 m×0.25 mm i.d. × 0.25  $\mu$ m dimensions. The carrier gas used was ultra high purity (UHP) Helium (99.999%) at a flow rate of 1.5ml min<sup>-1.</sup> The sample was injected in a split mode with split ratio 1:10. The injector temperature was maintained at 250 °C. The column temperature was programmed at 170°C (1 min hold) to 250°C (20°C min<sup>-1.</sup> The ion source temperature was 200° C and the interface temperature was 275°C. The mass detector was of quadrapole mass analyzer type in EI mode at an ionization voltage of 70 eV for ionization. Residues were estimated in SIM (Selected Ion Monitoring) mode in mass range of 272 m/z. The solvent delay was 2.5 min and retention time of spiromesifen under this condition was 9.0 minutes.

#### 3.4.4.2 Acetamiprid

#### Extraction

Chilli samples (50 g) obtained from control plots was chopped and then ground to a fine paste. A representative sample (10 g) was taken after quartering and was then spiked with analytical standard of acetamiprid (99% purity) at 0.01, 0.1, 0.25 and 2.5 mg kg<sup>-1</sup> level. It was further homogenized and extracted with 100 ml acetone. The extract was filtered through a glass funnel plugged with cotton. Extraction was repeated two times with 50 ml acetone each time. The combined filtrate was concentrated to 5 ml using rotary vacuum evaporator. The extract was taken in 500 ml separatory funnel and 100 ml saturated sodium chloride solution was added to it. The extract was partitioned with 50 ml hexane. The hexane layer was discarded. The extract was finally partitioned with dichloromethane (100 ml + 50 ml +50 ml) three times. The combined dichloromethane layer was collected after passing through anhydrous sodium sulphate. The extract was concentrated on rotary vacuum evaporator to about 5 ml and subjected to clean up for removal of coextractives and impurities.

#### Clean Up

The extract was cleaned up by column chromatography using activated florisil PR (6g) sandwiched between two layers of anhydrous sodium sulphate (3g) in a glass column having 2.2cm internal diameter and 60 cm length. The column was pre-washed using 20 ml hexane and extract was eluted with100 ml of hexane: acetone (1:1) mixture. The cleaned eluate was concentrated to near dryness in a rotary vacuum evaporator and the volume was made up to 5 ml with acetone for GC analysis.

# Gas Liquid Chromatograph Analysis

The cleaned extracts were analysed on gas liquid chromatograph, Shimadzu, Model – GC 2010 equipped with Ni<sup>63</sup> electron capture detector ( E CD) fitted with capillary column (J &W, DB-1) of 30 m×0.25 mm i.d. × 0.25  $\mu$ m dimension. The sample was injected in a split mode with split ratio 1:10. The injector and detector temperature was maintained at 250 °C and 300°C respectively. The column temperature was programmed at 160 °C to 270 °C at the rate of 5°C per minute (8 min hold). Ultra high purity (UHP) nitrogen was used as carrier gas with flow rate of 1.5ml min<sup>-1</sup> and linear gas velocity of 39.9 cm sec<sup>-1</sup>.The retention time of acetamiprid was 16.2 minutes.

#### 3.4.4.3 Imidacloprid

#### Extraction

Chilli samples (50 g) obtained from control plots was chopped and then ground to a fine paste. A representative sample (10 g) was taken after quartering and was then spiked with analytical standard of imidacloprid (99.99% purity) at 0.01, 0.05 and 0.1 mg kg<sup>-1</sup> level. The sample was homogenized and extracted thrice using 30 ml of acetonitrile by shaking for thirty minutes in a mechanical shaker. The extracts were combined, concentrated to 10 ml under vacuum at  $40^{\circ}$  C using rotary vacuum flash evaporator. The concentrated extract was taken in a 500ml seperatory funnel and 100ml of saturated sodium chloride solution was added. The extract was then partitioned thrice using 25ml hexane each time and the organic layers were discarded. The aqueous layer was again partitioned with 25 ml of hexane: ethyl acetate (98:2) mixture and the organic phase were discarded. The aqueous phase was extracted thrice using dichloromethane (50 ml each time) and the combined extract was collected after passing through anhydrous sodium sulphate. This was again concentrated to 5ml in a rotary vacuum flash evaporator.

# Clean Up'

The extract was cleaned up by column chromatography using activated florisil PR (5g) sandwiched between two layers of anhydrous sodium sulphate (2-3g) in a glass column having 2.2cm internal diameter and 60 cm length. The column was pre washed with 30 ml ethyl acetate and the concentrated extract was eluted with 30 ml of acetonitrile. The eluate was concentrated near to dryness in a rotary vacuum flash evaporator and the residues were dissolved in 1ml acetonitrile (HPLC grade) and injected into High Performance Liquid Chromatography (HPLC) analysis.

# High Performance Liquid Chromatography (HPLC) analysis.

The residues of imidacloprid were estimated by high performance liquid chromatography (HPLC), Model Schimadzu LC 20AT, reverse phase (RP) with Phenomenex C-18 column and PDA detector. The mobile phase was acetonitrile: water (35:65, v/v) with a flow rate of 1ml min<sup>-1</sup> flow rate. Detection was at a wavelength of 270 nm with an injection volume of 20  $\mu$ l (fixed loop). The retention time of imidacloprid was 5.96 minutes.

# 3.4.4.4 Propargite

## Extraction

Chilli samples (50 g) obtained from control plots was chopped and then ground to a fine paste. A representative sample (10 g) was taken after quartering and was then spiked with analytical standard of propargite at 0.01, 0.05 and 0.1 mg kg<sup>-1</sup> level. Chilli samples was further homogenized was extracted with acetone 100ml. The extract was filtered into 1 L seperatory funnel along with rinsing of acetone. The acetone extract was again diluted with 200 ml of 10 % sodium chloride solution and partitioned thrice using 75ml dichloromethane solution. The dichloromethane fractions were combined and passed through anhydrous sodium sulphate.

#### **Clean Up**

The extract was cleaned up by treating with 180 mg of activated charcoal powder for about 2-3 hours at room temperature. The clear extracts so obtained were filtered through Whatman filter paper No. 1 concentrated to near dryness and added about 5ml acetonitrile (HPLC grade) and again concentrated using rotary vaccum evaporator. This process was repeated to completely evaporate dicloromethane and final volume was reconstituted to about 5 ml using HPLC grade acetonitrile.

# High Performance Liquid Chromatography (HPLC) analysis.

The instrument parameters for HPLC analysis of propargite is same as described in3.4.4.3. The retention time of propargite was 13.29 minutes.

#### 3.4.4.5 Ethion

#### Extraction

Chilli samples (50 g) obtained from control plots was chopped and ground to a fine paste. A representative sample (10 g) was taken after quartering and was then spiked with analytical standard of ethion at 0.01, 0.1, 0.25 and 0.5 mg kg<sup>-1</sup> level. The ground samples were further homogenized was extracted with acetone 50ml. The extract was filtered through glass wool plugged in a filter funnel. The residual pulp was re-extracted with 50 ml of acetone and filtered. These extracts were combined, transferred to a seperatory funnel of 1 litre capacity and diluted with 200 -300 ml of 5% sodium chloride solution. The extract was then partitioned with 100, 50 and 50 ml of dichloromethane. The combined dichloromethane extracts thus obtained were concentrated to 2-3ml in vacuum using a rotary vacuum evaporator.

# Clean Up

The extracts were cleaned up by column chromatography using a mixture of silica gel (60-120 mesh) and activated carbon as an adsorbent. A glass column (2cm diameter  $\times$  60 cm long) was packed with activated silica gel (20 g + 0.2g charcoal) in between two layers of anhydrous sodium sulphate. The column was pre- washed with dichloromethane following which the concentrated extract was poured over it. The extract was eluted with a freshly prepared solvent mixture of dichloromethane: acetone (1:1 v/v). The eluate was concentrated to near dryness in a rotary evaporator under vacuum and then transferred to 5ml acetone for further analysis.

#### Gas Liquid Chromatograph Analysis

The instrument parameters for GLC analysis of ethion is same as described in 3.4.4.2. The retention time of ethion is 14.80 minutes.

## 3.4.4.6 Dimethoate

#### Extraction

Chilli samples (50 g) obtained from control plots was chopped and ground to a fine paste. A representative sample (10 g) was taken after quartering and was then spiked with analytical standard of dimethoate at 0.01, 0.1, 0.5 and 1 mg kg<sup>-1</sup> level. Samples were further homogenized and extracted with (2×100 ml portions) acetone in a high speed blender. The contents were filtered through Buchner funnel using filter paper No 1. The filtrates were combined and evaporated to about 20 ml using a rotary vacuum evaporator and transferred to separatory funnel. The extract was diluted with 200 ml (2×100) of hexane and dichloromethane (1:1) mixture. After shaking, the aqueous layer was again diluted with 30 ml sodium chloride solution and partitioned twice (2×50ml) with dichloromethane (2×50 ml). The extract was passed through anhydrous sodium sulpahate for removal of traces of moisture and the combined dichloromethane extracts thus obtained were concentrated to 2-3ml under vacuum using a rotary vacuum evaporator.

# Clean up

The extracts were cleaned up by column chromatography using florisil as an adsorbent. A glass column (2cm diameter  $\times$  60 cm long) was packed with activated florisil PR (4g) in between two layers of anhydrous sodium sulphate (2g). The column was pre- washed with 10ml of hexane following which the concentrated extract was poured over it. The extract was eluted with a freshly prepared solvent mixture of 100 ml hexane: acetone (9:1 v/v). The eluate was concentrated to near dryness in a rotary evaporator under vacuum and then made up to 5ml with hexane for further analysis.

# Gas Liquid Chromatograph Analysis

The instrument parameters for GLC analysis of dimethoate is same as described in 3.4.4.2. The retention time of dimethoate is 5.61 minutes.

## 3.4.4.7 Oxy demeton methyl

#### Extraction

Chilli samples (50 g) obtained from control plots was chopped and ground to a fine paste. A representative sample (10 g) was taken after quartering and was then spiked with analytical standard of oxydemeton methyl at 0.03, 0.1 and 0.5 mg kg<sup>-1</sup> level. Sample was homogenized and extracted sequentially thrice using 30 ml acetonitrile by shaking for thirty minutes in a mechanical shaker. The extracts were combined, concentrated to 10 ml under vacuum at 40°C using rotary vacuum flash evaporator and 90 ml of distilled water containing 10g sodium chloride was added. The aqueous layer was partitioned once using 10 ml hexane and the organic layer was discarded. The aqueous phase was then extracted thrice using 25 ml dichloromethane and the lower organic phase was collected through 10 g anhydrous sodium sulphate.

The combined dichloromethane extract was concentrated under vacuum in a rotary vacuum flash evaporator to 5 ml.

# Clean Up

The concentrated extract was passed through a chromatographic column loaded with 5g activated florisil sandwiched between two layers of anhydrous sodium sulphate (4 g each). The column was pre-washed with 1:1 hexane: acetone and the concentrated extract was loaded on the column and eluted with 50 ml hexane: acetone (1:1) mixture. The eluate was concentrated to 5 ml and the residue was oxidized using 25ml potassium permagnate. After 30 minutes, it was partitioned thrice using 50 ml chloroform and the lower organic phase was collected. The combined chloroform extract was concentrated under vacuum in a rotary vacuum flash evaporator to 1ml and the residues estimated using GC-MS.

# Gas Chromatograph – Mass spectrometric Analysis

The cleaned extracts were analysed on Shimadzu GC-MS2010 QP plus equipped with Mass Detector fitted with capillary column (J &W, DB-1 MS ultra innert) of 30 m X 0.25 mm i.d. X 0.25  $\mu$ m dimensions. The ultra high purity (UHP) Helium (99.999%) was used as carrier gas at a flow rate of 1.5ml min<sup>-1.</sup> The sample was injected in a split mode with split ratio 1:10. The injector temperature was maintained at 250 °C. The column temperature programme is given below:-

Rate in <sup>o</sup> C	Temperature	Hold time
	. 100	0
15	160	0
8	206	0
2	220	0
10	260	10

60

The total programme time was 30.75 minutes. The mass fragmentation was done in EI mode at an ionization voltage of 70 eV. Residues were estimated in SIM (Selected Ion Monitoring) mode. The m/z (mass/charge ratio) used for quantitation was 169. The retention time of Demeton-S- methyl sulfon is 10.53 minutes.

# 3.4.5 Sampling

The chilli plants were sprayed twice with the above insecticides at seven days intervals during the fruiting stage. Sprayed sample of green chilli fruit were collected from each plot 0 (2hrs after application), 1, 3, 5, 7, 10, 15, 21 and 30 days after the final spraying. 500g sample of chilli fruits harvested from each replicate of both the treatment and control plots and were brought to laboratory for further processing.

# 3.4.6 Analytical procedure

The laboratory procedures for pesticide residue analysis of chilli fruits were described in 3.4.4

#### 3.4.8 Data Analysis

The persistence of insecticide is generally expressed in terms of half life ( $DT_{50}$ ), time for disappearance of pesticide to 50% of its initial concentration. The half life ( $DT_{50}$ ) as well as time required to reach below tolerance level ( $T_{tol}$ ) were calculated using Hoskins formula (Hoskin, 1961).

# 3.5 STANDARDISATION OF COMMON PRACTICES TO DECONTAMINATE PESTICIDE RESIDUES FROM CHILLI FRUITS

A third spray application of insecticides listed under section 3.3.1 except oxy demeton methyl was given to study this aspect. Green chilli fruits were collected at 0 and  $5^{\text{th}}$  days after application of pesticides in the experiment described under section 3.3 was subjected to different

decontaminating solutions to assess the extent of removal of residues from them. The different decontaminating solutions used in this experiment are detailed below.

- Common salt 2% (20 g of common salt dissolved in one litre water)
- Tamarind 2% (20 g of preserved tamarind pulp extracted in one litre water)
- Lemon juice 2% (20 ml of fresh lemon juice diluted in one litre water)
- Lemon juice + common salt 2% (20 ml of fresh lemon juice and 20 g of common salt diluted in one litre of water).
- Vinegar 2% (20 ml of vinegar diluted in one litre water)
- Curd 2% (20 ml of curd diluted in one litre of water)
- Slaked lime 2% (20 g of hydrated lime dissolved in one litre of water)
- Baking soda 2% (20 g of baking soda powder dissolved in one litre of water)

# 3.5.1 Sample Preparation

100 g of chilli fruits were dipped individually in these treatment solutions for twenty minutes followed by washing in water. Chilli samples were then homogenized after chopping into small pieces and the representative sample (10g) was used for residue estimation. The analytical procedure for residue estimation of the six different insecticides was followed as detailed under section 3.4.4.1 to 3.4.4.6 of experiment 3.3 except for imidacloprid and propargite. These two compounds were analyzed using LC-MS/MS and the instrument parameters of the analysis are given below.

# Liquid Chromatography Mass spectrometric – Mass spectrometric (LCMS-MS) Analysis

The residues of imidacloprid and propargite were estimated using Ultra Performance Liquid Chromatograph – Tandem Mass Spectrometer (Triple Quadrapole) UPLC – MS/MS system.

#### **UPLC Analysis**

UPLC separations were carried out on Aquity UPLC system using a reverse phase column Aquity UPLC BEH  $C_{18}$  (2.1 × 50 mm) with 1.7 µm spherical porous particles. The elution was performed using gradient water and acetonitrile. Total run time was ten minutes with a flow rate of 0.5ml/minute. Injection volume of 5 µl was used in all experiments.

#### **MS/MS** Analysis

The instrument was operated in positive ESI mode. The ESI parameters as well as selection and tuning of MS/MS transitions and analyte dependent parameters (Collision energy (C.E), Declustering potential (D.P)). were performed by direct infusion of pesticide individual standard solutions into the mobile phase flow (50: 50, v/v, water : acetonitrile 0.5ml / minute). In all experiments the following parameters were used.

Source temperature ·	-	500°C
Curtain gas	-	10
Ion spray voltage	-	5500 V

The retention time of imidacloprid and propargite are 1.33 and 3.25 minutes respectively.

The percentage reduction of residues was worked out by comparing the residue data in processed fruits with unprocessed fruits.

# 3.5.2 Processing Factor

The processing factor was calculated by using the following formula

Residue in washed chilli fruits

Processing factor = Residue in unwashed chilli fruits

# RESULTS

# 4. RESULTS

# 4.1 LABORATORY EVALUATION OF NEW GENERATION INSECTICIDES AGAINST CHILLI MITE AND THRIPS.

The results on the efficacy of new generation insecticides against three major sucking pests of chilli viz. P. latus, S. dorsalis and A. gossypii when evaluated in the laboratory and pot culture study are presented in Table 4 to 6.

4.1.1 P. latus

Contact toxicity of new generation insecticides to *P. latus* was evaluated in the laboratory by leaf disc method and the data are presented in Table 4.

Among the various new generation insecticides evaluated, spiromesifen 100 g a.i. ha<sup>-1</sup> and acetamiprid 20 g a.i. ha<sup>-1</sup> recorded 100 per cent mortality of chilli mite and were superior to other insecticides at 24hrs after release (Table 4). Spinosad 75 g a.i. ha<sup>-1</sup> (77.8), imidacloprid 20 g a.i. ha<sup>-1</sup> (77.8), thiamethoxam 40 g a.i. ha<sup>-1</sup> (61.11), and propargite 570 g a.i ha<sup>-1</sup>(72 .22) followed the two superior treatments indicated above and were on par with each other. These were followed by dimethoate 300 g a.i. ha<sup>-1</sup> (44.4), indoxacarb 60 g a.i. ha<sup>-1</sup> (38.89) and spirotetramat 60 g a.i. ha<sup>-1</sup> (27.78) which were on par with each other. Flubendiamide 60 g a.i. ha<sup>-1</sup> (11.11) recorded the least mortality after 24 hours.

At 48 hours after release of the mites, spinosad and propargite also recorded 100 per cent mortality of chilli mite along with spiromesifen and acetamiprid. Imidacloprid (91.67) was the next best treatment and all these five treatments were found on par with each other. These were followed by

[ ·		Per cent n	nortality obse	rved at differ	rent intervals	after release	
	Dosage		P. latus		S. dorsal	is	
Treatments	(g a.i.	24hrs	48 hrs	72 hrs	24hrs	48hrs	72hrs
	ha <sup>-1</sup> )						
	75	77.78	100.00	100.00	83.33	94.40	100.00
Spinosad		(66.57)	(88.83)	(88.83)	(69.82)	(81.18)	(88.83)
Spiramogifan	100	100.00	100.00	100.00	83.33	100.00	100.00
Spiromesifen		(88.83)	(88.83)	(88.83)	(69.82)	(88.83)	(88.83)
Spirotetamat	. 60	27.78	66.67	91.67	50.00	77.78	83.33
Sphotetalinat		(31.54)	(55.21)	(79.22)	(44.99)	(66.57)	(69.82)
Indoxacarb	60	38.89	61.11	86.67	50.00	55.55	72.22
hidoxacaro		(38.03)	(51.49)	(76.14)	(44.99)	(48.24)	(58.46)
Imidacloprid	20	77.78	91.67	94.40	94.44	94.44	100.00
midacioprid		(66.57)	(79.22)	(81.19)	(81.18)	(81.18)	(88.83)
Thiamethoxam	40	61.11	83.33	93.33	94.44	100.00	100.00
Thanceloxum		(51.96)	(69.82)	(80.36)	(81.18)	(88.83)	(88.83)
Flubendiamide	60	11.11	28.34	33.33	22.22	50.0	61.11
Tubbindiumide		(16.45)	(31.10)	(34.78)	(27.82)	(44.99)	(51.49)
Acetamiprid	20	100.00	100.00	100.00	100.00	100.00	100.00
Accumpto		(88.83)	(88.83)	(88.83)	(88.83)	(88.83)	(88.83)
Propargite	<sup>-</sup> 570	72.22	100.00	100.00	66.67	72.22	100.00
Tiopargne		(58.45)	(88.83)	(88.83)	(54.74)	(58.46)	(88.83)
Dimethoate	300	44.44	86.67	100.00	94.44	100.00	100.00
1		(41.75)	(76.14)	(88.83)	(81.18)	(88.83)	-(88.83)
CD(0.05)		(21.803)	(16.248)	(19.269)	(19.878)	(16.202)	(10.433)
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Table 4. Mortality of *Polyphagotarsonemus latus* and *Scirtothrips dorsalis* observed at different intervals when exposed to insecticide treated leaf discs of chilli.

Figures in the parentheses are angular transformed values.

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dimethoate (86.67), thiamethoxam (83.33) which were at par. Spirotetramat (66.67) and indoxacarb (61.11) followed the above treatments in their efficacy against mites which were on par with each other and were found superior over flubendiamide which recorded a mortality of 28.34 per cent only at 48 hrs after release.

At 72 hours after release, dimethoate also recorded 100 % mortality in addition to spinosad, spiromesifen, acetamiprid and propargite. These treatments were followed by thiamethoxam (93.3), imidacloprid (94.4), spirotetramat (91.67), and indoxacarb (86.67). All the above treatments were found at par with each other, where as flubendiamide recorded the lowest mortality (33.33).

#### 4.1.2 S. dorsalis

The data on the contact toxicity of pesticides to the  $2^{nd}$  instar nymphs of *S. dorsalis* is presented in Table 4. In the observations recorded at 24 hours after application of the recommended dose of pesticides, 22.22 to 100 per cent mortality were recorded in the different treatments. Hundred per cent mortality of thrips was recorded in acetamiprid treated leaf disc and it was followed by thiamethoxam (94.44), dimethoate (94.44), imidacloprid (94.44) spinosad (83.33) and spiromesifen (83.33) and these treatments were on par with each other. Propargite (66.67) was the next best treatment followed by spirotetramat and indoxacarb which recorded only an average mortality of 50.00 per cent. As in the case of the chilli mite, lowest mortality of thrips was recorded in flubendiamide treated leaf disc (22.22).

The insecticides which achieved 100 per cent mortality after 48 hours of release included dimethoate, spiromesifen and thiamethoxam in addition to acetamiprid which recorded cent per cent mortality after 24 hours. These were followed by spinosad (94.4) and imidacloprid (94.4) and all the above treatments were found on par with each other. Spirotetramat (77.78) and propargite (72.22) were the next best treatments followed by indoxacarb and flubendiamide which recorded 55.55 and 50.00 per cent mortality respectively.

At 72 hours after release of thrips into the insecticide treated discs, all the insecticide treatments except spirotetramat (83.33), indoxacarb (72.22) and flubendiamide (61.11) recorded cent per cent mortality.

#### 4.2 POT CULTURE STUDY

A pot culture experiment was conducted to study the efficacy of new generation insecticides in comparison with conventional insecticides on the nymph and adults of *P. latus*, adults of *S. dorsalis* and the intensity of leaf curl caused by them under conditions of natural infestation. Data are presented in Table 5.

#### 4.2.1 *P. latus* (Nymph)

Considering the effect of various new generation insecticides on the nymphs of *P. latus* at one day after spraying (DAS), propargite stood first in recording the lowest mite population (0.30 / leaf) and was found on par with other treatments *viz.*, dimethoate (1.64 / leaf), imidacloprid (1.74 / leaf) and acetamiprid (1.98 / leaf). This was followed by spiromesifen (2.22 / leaf), spinosad (2.85 / leaf), indoxacarb (3.39 / leaf) and spirotetramat (4.13 / leaf), respectively and were on par in their efficacy against nymphs of chilli mite. The highest population of mite nymphs was recorded in flubendiamide sprayed plants (6.18 / leaf) which was significantly higher than all other treatments but found on par with those in untreated chilli plants (6.66 / leaf).

At 3DAS, lowest incidence of chilli mites was recorded in propargite treated chilli plants (0.30 / leaf) followed by imidacloprid, spiromesifen, acetamiprid and dimethoate which all recorded a mean population of 0.63 nymphs per leaf. This was followed by indoxacarb (1.74 / leaf) thiamethoxam (2.32 / leaf) and spinosad (2.61 / leaf) and all the treatments were on par.

	Dosage			Mean	population	of P latus	per leaf			Mean p	Mean population of S.dorsalis per leaf				LCI	
	(g	Nymph Adult												2 weeks	4 weeks	
Treatment	a.i./ha)	IDAS	3DAS	5DAS	7DAS	IDAS	3DAS	5DAS	7DAS	IDAS	3DAS	5DAS	7DAS	after spraying	after spraying	
Spinosad	75	2.85 (1.962)	2.61 (1.901)	1.59 (1.609)	1.21 (1.488)	5.25 (2.499)	4.18 (2.276)	1.94 (1.715)	0.63 (1.276)	1.33 (1.526)	0.67 (1.292)	0.33 (1.153)	0.00 (1.000)	1.67	1.00	
Spiromesifen	100	2.22 (1.794)	0.63 (1.276)	0.00 (1.000)	0.00 (1.000)	0.91 (1.380)	0.91 (1.380)	0.00 (1.000)	0.00 (1.000)	1.67 (1.634)	1.33 (1.526)	0.67 (1.292)	0.00 (1.000)	0.67	0.00	
Spirotetamat	60	4.13 (2.265)	3.62 (2.149)	3.40 (2.098)	2.96 (1.989)	5.84 (2.616)	4.66 (2.378)	3.00 (2.000)	1.94 (1.715)	2.33 (1.824)	1.67 (1.634)	1.33 (1.526)	1.33 (1.526)	2.00	1.33	
Indoxacarb	60	3.39 (2.097)	1.74 (1.656)	1.39 (1.516)	0.55 (1.244)	5.98 (2.641)	3.27 (2.068)	2.55 (1.883)	1.00 (1.414)	2.67 (1.915)	2.33 (1.824)	1.67 (1.634)	1.33 (1.526)	2.30	1.00	
Imidacloprid	2Ō	1.74 (1.656)	0.63 (1.276)	0.63 (1.276)	0.29 (1.136)	0.91 (1.382)	0.78 (1.33)	0.54 (1.244)	0.00 (1.000)	1.00 (1.414)	0.67 (1.292)	0.33 (1.153)	0.00 (1.000)	1.00	0.33	
Thiamethoxam	40	3.32 (2.078)	2.32 (1.821)	1.31 (1.521)	0.30 (1.140)	4.48 (2.341)	2.65 (1.910)	1.17 (1.471)	0.55 (1.244)	1.33 (1.526)	1.33 (1.526)	1.00 (1.414)	0.33 (1.153)	1.33	1.00	
Flubendiamide	60	6.18 (2.681)	3.40 (2.098)	1.94 (1.715)	1.32 (2.515)	5.61 (2.570)	4,91 (2.431)	4.59 (2.365)	3.97 (2.228)	3.33 (2.080)	2.67 (1.915)	2.33 (1.824)	2.00 (1.732)	2.33	2.00	
Acetamiprid	20	1.98 (1.727)	0.63 (1.276)	0.29 (1.138)	0.00 (1.000)	2.96 (1.989)	1.49 (1.577)	0.00 (1.000)	0.00 (1.000)	0.33 (1.153)	0.00 (1.000)	0,00 (1.000)	0.00 (1.000)	1.33	1.00	
Propargite	570	0.30 (1.140)	0.30 (1.140)	0.00 (1.000)	0.00 (1.000)	0.78 (1.333)	0,63 (1.276)	0.00 (1.000)	0.00 (1.000)	2.33 (1.824)	1.67 (1.634)	1.33 (1.526)	1.33 (1.526)	1.33	0.00	
Dimethoate	300	1.64 (1.626)	0.63 (1.276)	0.30 (1.138)	0.00 (1.000)	2.96 (1.989)	1.49 (1.577)	0.30 (1.140)	0.30 (1.140)	1.33 (1.526)	1.00 (1.414)	0.67 (1.292)	0.00 (1.000)	1.67	1.00	
Control	Water spray	6.66 (2.767)	7.33 (2.886)	7.66 (2.942)	8.00 (3.00)	8.32 (3.05)	8.38 (3.063)	9.30 (3.210)	10.55 (3.398)	5.00 (2.449)	5.33 (2.515)	6.00 (2.645)	6.00 (2.645)	3.33	3.66	
CD		(0.6106)	(0.8029)	(0.8140)	(0.6572)	(0.8108)	(0.7903)	(0.6070)	(0.4893)	(0.4410)	(0.2842)	(0.3519)	(0.2031)	0.884	0.834	

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Table 5. Mean population of Polyphagotarsonemus latus, Scirtothrips dorsalis and the leaf curl index (LCI) in potted chilli plants.

Figures in the parenthesis are  $\sqrt{x+1}$  values, DAS - Days after spraying.

Among the insecticide treated plants, highest nymph population was recorded in spirotetramat (3.62 / leaf) and flubendiamide (3.40 / leaf), but it was significantly lower than untreated plants which recorded a population as high as 7.33 mite nymph per leaf.

At five days after spraying, no mite nymphs were recorded on spiromesifen and propargite treated chilli plants indicating them as the best treatments and was found on par with treatments like acetamiprid (0.29 / leaf), dimethoate (0.3 / leaf), imidacloprid (0.63 / leaf), thiamethoxam (1.31 / leaf), indoxacarb (1.39 / leaf) and flubendiamide (1.94 / leaf). Plants treated with spirotetramat had the highest nymph population (3.4 / leaf) when observed at 5 days after spraying, but was found significantly superior over the untreated potted plants which recorded a population of 7.66 nymphs / leaf.

The mite nymph population in almost all treatments drastically declined at the seventh day of spraying where the treatments like propargite, acetamiprid, dimethoate and spiromesifen were devoid of mite nymphs. These were followed by imidacloprid (0.29 / leaf), thiamethoxam (0.30 / leaf), indoxacarb (0.55 / leaf), spinosad (1.21 / leaf) and flubendiamide (1.32 / leaf) and all the above treatments were on par in their efficacy in suppressing the population of chilli mite nymphs. An average of more than two nymph per leaf was recorded in and spirotetamat (2.96 / leaf), where as the untreated chilli plants recorded a mean population of 8.00 nymps / leaf.

# 4.2.2 P. latus (Adult)

It was observed that one day after treatment, the lowest adult mite incidence was recorded in plants sprayed with propargite (0.78), spiromesifen (0.91) and imidacloprid (0.91). These were followed by acetamiprid and dimethoate which recorded an average of 2.96 mites per leaf and all the above treatments were on par. Thiamethoxam (4.48), spinosad (5.25) flubendiamide (5.61) and indoxacarb (5.98) were the next best treatments in the order of

efficacy and the treatments were on par and were significantly superior over untreated control (8.32).

At 3 DAS, the adult mite population followed the same trend as that in 1 DAS where propargite, imidacloprid and spiromesifen were the treatments which recorded the lowest mite population of 0.63, 0.78 and 0.91 per leaf respectively. The mite population declined uniformly in acetamiprid and dimethoate treated plants which recorded an average of 1.49 mites per leaf. Thiamethoxam (2.65) showed a medium level of control of adult mites followed by indoxacarb (3.27). All the above mentioned treatments were at par. Spinosad (4.18), spirotetramat (4.66) and flubendiamide (4.91) were on par in their efficacy and significantly superior over the untreated plants which recorded 8.38 mites per leaf.

At 5 DAS, no adult mite population was recorded on spiromesifen, propargite and acetamiprid treated chilli plants, whereas dimethoate, imidacloprid and thiamethoxam treated plants recorded 0.30, 0.54, 1.17 mites per leaf respectively. All the above treatments were found on par with each other. Spinosad (1.94), indoxacarb (2.55), spirotetramat (3.00) were the next best treatments. Flubendiamide sprayed chilli plants recorded maximum mites (4.59 / leaf) among the different insecticidal treatments but was significantly less compared to the population in the untreated plants (9.30).

The adult mite population reduced drastically at 7 DAS and no adult mites were recorded in spiromesifen, imidacloprid, acetamiprid and propargite treated chilli plants. These insecticidal treatments were at par with dimethoate (0.30), thiamethoxam (0.55), spinosad (0.63) and indoxacarb (1.00). Spirotetramat (1.94) and flubendiamide (3.97) were the next best treatments and were statistically superior over the untreated control (10.55).

# 4.2.3 S. dorsalis

When the data on the population of *S. dorsalis* was analysed at one day after spraying (Table 5), acetamiprid treated plants had the lowest population of thrips (0.33) and was on par with imidacloprid (1.00), spinosad (1.33), thiamethoxam (1.33) and dimethoate (1.33) treated plants. Spiromesifen (1.67), propargite (2.33) and spirotetramat (2.33) and indoxacarb (2.67) were the next best treatments in the order of their efficacy. As same as in the case of chilli mites, flubendiamide (3.33) was the least effective insecticide against thrips also, but was significantly superior to the untreated control plants which recorded an average population of five thrips per leaf.

Acetamiprid treated plants had no thrips population in the leaves sampled and was statistically superior over all the other insecticidal treatments at three days after spraying. Spinosad and imidacloprid had an average of 0.67 thrips per leaf followed by dimethoate (1.00) which were found on par. Spiromesifen (1.33), thiamethoxam (1.33), propargite (1.67), spirotetramat (1.67) and indoxacarb (2.33) were ranked equal in their efficacy against chilli thrips. Flubendiamide (2.67) recorded maximum population of thrips but was significantly superior to untreated plants (5.33).

At five days after spraying, the thrips population followed a similar trend as in the third day of spraying where acetamiprid treated chilli plants were devoid of chilli thrips population and was found on par with spinosad (0.33), imidacloprid (0.33), spiromesifen (0.67) and dimethoate (0.67). Thiamethoxam recorded an average of one thrip per leaf followed by propargite (1.33), spirotetramat (1.33) indoxacarb (1.67) and flubendiamide (2.33). All these treatments were significantly superior over untreated plants which recorded an average of 6 thrips per leaf.

At seven days after spraying, along with acetamiprid, dimethoate, spinosad, spiromesifen and imidacloprid also recorded no thrips population in the treated plants. Thiamethoxam sprayed chilli plants recorded an average of 0.33 thrips per plantand was found at par with the above insecticidal treatments whereas spirotetramat, indoxacarb and propargite treated plants had an average of 1.33 thrips per plant and flubendiamide treated plants had an average of 2 thrips per plant. All these treatments were significantly superior to untreated control plants (6.00).

#### 4.2.4 Leaf Curl Index (LCI)

Intensity of leaf curl caused by *P. latus, S. dorsalis* or both was assessed in terms of LCI worked out as per standard procedure for scoring and mean score presented in Table 5.

At two weeks after the insecticidal spraying in chilli plants, plants sprayed with spiromesifen had the lowest leaf curl index 0.67 followed by imidacloprid (1.0). Treatments done with thiamethoxam, acetamiprid, and propargite recorded an average LCI of 1.33 and was found on par with the above two treatments. Spinosad and dimethoate which recorded an average index of 1.67 followed by spirotetramat, indoxacarb and flubendiamide which recorded mean LCI values of 2, 2.3 and 2.33 respectively where as the untreated chilli plants recorded the maximum intensity of leaf curl (3.33).

At four weeks after spraying, leaf curl index followed the same trend as that observed in the preceding weeks after spraying where no leaf curling was recorded on spiromesifen and propargite treated plants. These were followed by imidacloprid (0.33), spinosad, thiamethoxam, acetamiprid and dimethoate all of which recorded an average LCI of 1. Spirotetramat (1.33) and indoxacarb recorded 1.00, where as flubendiamide recorded the highest LCI (2.0) among different insecticidal treatments. The untreated plants recorded an average LCI of 3.66.

# 4.3 LABORATORY EVALUATION OF NEW GENERATION INSECTICIDES ON CHILLI APHIDS.

#### 4.3.1 A. gossypii

The results of the pot culture study (Table 6) to evaluate the effect of different new generation insecticides on chilli aphids, *A. gossypii* revealed that thiamethoxam (100), acetamiprid (100) and imidacloprid (98.67) were highly toxic to chilli aphids, as these insecticides caused total mortality of aphids at one day after spraying. Dimethoate (85.33) and spirotetramat (80.00) were the next best treatments. Indoxacarb showed moderate toxicity by reducing the aphid population to the tune of 65.33 per cent. All the other insecticides, *viz.* spiromesifen (28), propargite (20) and flubendiamide (13.33) recorded low mortality at 24 hours after spraying.

At two days after spraying, the mortality of aphids followed the same trend as in the case of one day after spraying, where thiamethoxam, acetamiprid and imidacloprid killed the aphid population completely followed by spirotetramat, which killed 93.33 per cent aphids and found as the next best treatment and the treatments were significantly different from others. Dimethoate and indoxacarb recorded 86.67 and 80.00 per cent mortality respectively and were found on par. Spiromesifen (78.67) was the next best treatment. Propargite, spinosad and flubendiamide recorded mortality less than 50% only, the values being viz, 45.33, 41.33 and 37.33 respectively.

Spiromesifen and dimethoate recorded cent per cent mortality at 3 DAS along with thiamethoxam, acetamiprid and imidacloprid. All the insecticides except flubendiamide (44.00) and spinosad (58.67) recorded more than 80.00 per cent mortality at 3 DAS *viz.* propargite (86.67), spirotetramat (98.67) and indoxacarb (98.67). Almost all the insecticides attained cent per cent mortality at 4 DAS except spinosad and flubendiamide which recorded a mortality of 73.33 per cent each.

Treatment	Dosage	% mortality observed at different intervals after release							
	(g a.i./ha)	1 DAS	2DAS	3 DAS	4DAS				
<u>}</u>									
Spinosad	75	18.67	-41.33	58.67	73.30				
-		(24.52)	(42.29)	(50.06)	(60.82)				
	100	20.00	70 67	100.00	100.00				
Spiromesifen	100	28.00 (31.91)	78.67 (63.26)	100.00 (88.83)	(88.83)				
		(51.91)	(05.20)	(00.05)	(00.05)				
Spirotetamat	60	80.00	93.33	98.67	100.00				
-		(63.74)	(77:55)	(85.77)	(88.83)				
T T	(0)	(5.22	00.00	00.67	100.00				
Indoxacarb	60	65.33	80.00	98.67	100.00 (88.83)				
		(54.21)	(63.74)	(85.77)	(00.03)				
Imidacloprid	20	98.67	100.00	100.00	100.00				
		(85.67)	(88.83)	(88.83)	(88.83)				
					1				
Thiamethoxam	40	100.00	100.00	100.00	100.00				
		(88.83)	(88.83)	(88.83)	(88.83)				
Flubendiamide	60	13.33	37.33	44.00	73.33				
		(16.32)	(37.62)	(41.52)	(59.01)				
Acetamiprid	20	100.00	100.00	100.00	100.00				
		(88.83)	(88.83)	(88.83)	(88.83)				
Propargite	570	20.00	45.33	86.67	100				
Tiopargree	0,0	(26.26)	(39.98)	(68.91)	(88.83)				
Dimethoate	300	85.33	86.67	100	100				
		(67.81)	(70.43)	(89.43)	(89.43)				
CD(0.05)		(13.960)	(8.653)	(6.864)	(8.723)				
		(13.500)	(0.055)		(0.725)				
	<u> </u>	1		·					

Table 6. Mortality of chilli aphids Aphis gossypii exposed to new generation insecticides.

Figures in the parentheses are angular transformed values.

# 4.4 LABORATORY EVALUATION OF NEW GENERATION INSECTICIDES ON THEIR SAFETY TO NATURAL ENEMIES.

# 4.4.1 Predatory mite (Amblyseius spp)

The data on the contact toxicity of natural enemies to the new generation insecticide molecules and conventional insecticides are presented in Table 7.

At 24 hours after treatment, less than 50 % mortality of the predatory mite, *Amblyseius* spp was observed in flubendiamide (28.87), spiromesifen (34.44), thiamethoxam (34.44) and indoxacarb (41.09) treated leaf discs confirming its safety to natural enemies. Moderate toxicity to predatory mite was observed in acetamiprid (52.22) and spirotetramat (58.89) treated leaf disc and were on par with imidacloprid and spinosad which recorded an average of 64.45 per cent mortality. Maximum mortality of predatory mite was observed in propargite (94.40) followed by dimethoate (82.21).

At 48 hours after release of predatory mite in to the insecticide treated leaf disc, flubendiamide (53.33), acetamiprid (57.77), indoxacarb (57.78), spiromesifen (63.32) thiamethoxam (64.45) and spinosad (70.01) recorded moderate level of toxicity to predatory mite and was found on par with imidacloprid which recorded an average mortality of 82.22 percentage. Dimethoate recorded 87.77 per cent mortality whereas hundred per cent mortality of the predatory mite was observed in propargite treated leaf disc.

Lowest mortality of predatory mite at 72 hours after release was noticed in acetamiprid (53.34) followed by spiromesifen (61.67), spirotetramat (68.31), thiamethoxam (68.33) and flubendiamide (78.33) and all the treatments were found at par with each other. More than eighty per cent mortality was recorded in indoxacarb (83.30), spinosad (83.53) and imidacloprid (84.99) treatments. Cent per cent mortality of predatory mite was noticed in dimethoate treated leaf disc along with propargite which recorded 100 per cent mortality after 48 hours of release.

Treatment	Dosage		Percentage mortality observed at different intervals after release on treated leaf disc/ leaf												
	(g a.i./ha)		Amblyseius sp			Coccinellid	grubs	Hemerobid larvae							
	<u> </u>	24hrs	48hrs	72hrs	24hrs	48hrs	72hrs	24hrs	48hrs	72hrs					
Spinosad	75	64.45 (53.89)	70.01 (61. <b>54</b> )	83.53 (74.23)	60.00 (51.46)	73.33 (59.2)	86.67 (71.86)	46.67 (43.08)	66.67 (54.99)	76.67 (65.79)					
Spiromesifen	100	34.44 (35.61)	63.32 (53.28)	61.67 (51.92)	0.00 (1.28)	20.00 (22.36)	20.00 (22.36)	20.00 (22.35)	40.00 (39.23)	38.33 (38.07)					
Spirotetamat	60	58.89 (50.17)	69. <b>9</b> 9 (57.13)	68.31 (56.14)	46.67 (43.07)	53.33 (46.92)	53.33 (46.92)	33.33 (35.09)	40.00 (39.23)	38.33 (38.07)					
Indoxacarb	60	41.09 (39.83)	57.78 (49.57)	83.3 (69.59)	46.67 (43.07)	60.00 (50.769)	73.33 (59.2)	40.00 (39.23)	53.30 (46.92)	51.67 (46.14)					
Imidacloprid	20	64.45 (53.43)	82.22 (65.006)	84.99 (70.75)	60.00 (51.145)	80.00 (63.44)	86.67 (71.86)	80.00 (67.64)	93.33 (80.29)	100.00 (88.83)					
Thiamethoxam	40	34.44 (42.09)	64.45 (53.42)	68.33 (56.14)	53.33 (47.3)	60.00 (51.14)	73.33 (59.2)	53.33 (46.92)	60.00 (50.77)	60.00 (50.769)					
Flubendiamide	60	28.87 (32.36)	53.33 (46.9)	78.33 (66.53)	33.33 (35.009)	40.00 (38.85)	40.00 (38.85)	20.00 (22.35)	33.33 (35.09)	38.33 (38.07)					
Acetamiprid	20	52.22 (46.32)	57.77 (49.55)	53.34 (46.95)	46.67 (43.07)	60.00 (51.14)	73.33 (59.2)	60.00 (50.77)	80.00 (63.43)	85.00 (70.71)					
Propargite	570	94.44 (81.19)	100.00 (88.83)	100.00 (88.83)	60.00 (50.769)	60.00 (51.14)	80.00 (63.44)	40.00 (39.23)	60.00 (50.76)	61.67 (51.92)					
Dimethoate	300	82.21 (65.07)	87.77 (72.69)	100.00 (88.83)	80.00 (63.44)	100.00 (88.83)	100.00 (88.83)	80.00 (63.44)	100.00 (88.83)	100.00 (88.83)					
CD(0.05)		(13.232)	(19.260)	(22.970)	(13.993)	(18.166)	(21.919)	(16.058)	(10.281)	(19.793)					

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Table 7. Mortality of Amblyseius spp, coccinellid grubs and hemerobid larvae exposed to insecticide treated leaf/leaf disc

Figures in the parentheses are angular transformed values.

# 4.4.2 Coccinellid Grubs

All the grubs released in to the spiromesifen treated chilli leaf were alive after 24 hours proving that it is highly safe to coccinellid grubs. Less than 50% mortality was observed in spirotetramat, indoxacarb, acetamiprid which recorded an average mortality of 46.67 and flubendiamide (33.33) recorded the lowest mortality of coccinellid grubs at 24 hours after release. Thiamethoxam recorded 53.30 per cent mortality of grubs followed by spinosad, imidacloprid and propargite which recorded an average mortality of 60 per cent. Significantly higher mortality of the coccnellid grubs was noticed in dimethoate (80) treated chilli leaf.

After 48 hours after release, the per cent mortality of coccinellids rose to 20 per cent in spiromesifen and was statistically superior to other insecticidal treatments in their safety to natural enemies. Flubendiamide which recorded 40 per cent mortality was the next best treatment. Spirotetramat (53.33) was found on par with Indoxacarb, thiamethoxam, propargite and acetamiprid which all recorded 60 per cent mortality of coccinellid grubs. Spinosad (73.33) and imidacloprid (80) were found to be more toxic to coccinellid grubs than the other insecticides. Cent per cent mortality of grubs was noticed only in dimethoate treatment.

In the observations recorded at 72 hours after release of the grubs, spiromesifen recorded only 20 per cent mortality and was statistically on par with flubendiamide which recorded a mortality of 40 per cent. Spirotetramat (53.33) indoxacarb (73.30) thiamethoxam (73.30) and propargite (80) were on par in their safety to natural enemies where as dimethoate proved to be significantly toxic to coccinellid grubs causing cent per cent mortality.

### 4.4.3 Hemerobid larvae

The different insecticidal treatments varied significantly with respect to in their toxicity to hemerobid larvae after 24 hours of treatment where spiromesifen and flubendiamide recorded lowest mortality of 20 per cent followed by spirotetramat (33.33), indoxacarb (40) and propargite (40).The next safer insecticides were spinosad (46.67), thiamethoxam (53.33) and acetamiprid (60). The highest mortality (80) after one day was recorded in imidacloprid and dimethoate treated leaves.

On the second day also, the same trend in safety was observed, where flubendiamide (33.33) was followed by spiromesifen and spirotetramat which recorded an average mortality of 40 per cent and were statistically on par. The next best treatments in the order of efficacy were indoxacarb (53.30), thiamethoxam (60) propargite (60) and spinosad (66.67). Acetamiprid (80) and imidacloprid (93.30) recorded high mortality of hemerobid larvae, but cent per cent mortality of hemerobid larvae was found only in dimethoate treatment.

After 72 hours of treatment, spiromesifen, spirotetramat and flubendiamide recorded 38.33 per cent mortality and were found on par with indoxacarb (51.67), thiamethoxam (60) and propargite (61.67). Significantly higher mortality was observed in spinosad (76.67) and acetamiprid (85) treated leaf, whereas imidacloprid caused 100 per cent mortality of larvae as in the case of dimethoate.

# 4.5 FIELD EVALUATION OF NEW GENERATION INSECTICIDES FOR THE MANAGEMENT OF CHILLI THRIPS AND MITES.

Based on the laboratory evaluation of contact toxicity to mite, thrips and aphids and safety evaluation against major predators, the new generation insecticides *viz*. spiromesifen, imidacloprid, thiamethoxam, acetamiprid, propargite, and three conventional insecticides *viz*, ethion, dimethoate and oxydemeton methyl as chemical check were selected for further evaluation in field. The results of the field evaluation are depicted in the tables 8 to 10.

# 4.5.1 First spray at vegetative stage of the crop at 30 days after transplanting (DAT)

## 4.5.1.1 Chilli mite (P. latus)

The mite population was lower during the period of first spraying done at 30 days after transplanting (Table 8). The propargite treated plots recorded the lowest mite population (0.20 / leaf) at one day after spraying and was on par with spiromesifen (0.22 / leaf), acetamiprid (0.26 / leaf) and imidacloprid (0.44 / leaf). The above treatments were statistically superior to all other insecticidal treatments. Thiamethoxam (0.62 / leaf), oxy demeton methyl (0.71)/ leaf), dimethoate (0.78 / leaf) and ethion (0.8 / leaf) were on par in their order of efficacy and significantly superior over the untreated plot which recorded an average mite population of 1.93 mites per leaf. At three days after spraying the mite population declined drastically in spiromesifen (0.06 / leaf) treated plots followed by acetamiprid and propargite which recorded an average of 0.13 / leaf, and were found significantly superior over all other treatments. The other insecticide treatments recorded mite population ranging from 0.24 to 0.42 per leaf. All the insecticidal treatments were found significantly superior over the untreated plants where the mite population increased to 2.16 / leaf from the first day after treatment. At five days after spraying, spiromesifen treated plots recorded 0.04 mites per plot and were on par with propargite and acetamiprid, which recorded an average mite population of 0.11 / leaf. Imidacloprid (0.13/leaf) and dimethoate (0.18/ leaf) were also found on par with the above treatments. All the other treatments recorded mite counts ranging from 0.27 to 0.28 / leaf and was found to be significantly superior over the untreated plants (2.48 / leaf). At seven days after spraying, no mite population was recorded in spiromesifen sprayed chilli plants and the treatment was on par with propargite (0.04 / leaf) and acetamiprid (0.04 / leaf) treated chilli plants.Imidacloprid, dimethoate and oxy demeton methyl plots recorded 0.18 mites per leaf and were on par with above treatments. The rest

Treatment	Dosage	L.*		Mite count/	leaf	Thrips count/leaf					
	g a.i.há <sup>-i</sup>	1 DAS	3 DAS	5 DAS	7 DAS	14DAS	1DAS	3 DAS	5DAS	7DAS	14DAS
Spiromesifen	100	0.22 (1.104)	0.06 (1.029)	0.04 (1.020)	0.00 (1.000)	0.37 (1.170)	0.30 (1.140)	0.17 (1.082)	0.00 (1.000)	0.05 (1.023)	0.13 (1.064)
Imidacloprid	20	0.44 (1.200)	0.24 (1.114)	0.13 (1.063)	0.18 (1.086)	0.82 (1.349)	0.34 (1.157)	0.20 (1.095)	0.02 (1.011)	0.11 (1.053)	0.22 (1.103)
Thiamethoxam	40	0.62 (1.273)	0.42 (1.191)	0.27 (1.127)	0.22 (1.104)	0.98 (1.407)	0.30 (1.140)	0.20 (1.095)	0.09 (1.043)	0.09 (1.043)	0.16 (1.077)
Acetamiprid	20	0.26 (1.122)	0.13 (1.063)	0.11 (1.053)	0.04 (1.020)	0.56 (1.249)	0.27 (1.127)	0.103 (1.050)	0.02 (1.011)	0.067 (1.032)	0.11 (1.053)
Propargite	570	0.20 (1.095)	0.13 (1.063)	0.11 (1.053)	0.04 (1.020)	0.38 (1.175)	0.71 (1.308)	0.45 (1.204)	0.27 (1.127)	0.35 (1.163)	0.42 (1.192)
Dimethoate	300	0.78 (1.334)	0.38 (1.174)	0.18 (1.086)	0.18 (1.086)	0.97 (1.403)	0.42 (1.192)	0.27 (1.127)	0.11 (1.053)	0.11 (1.053)	0.26 (1.123)
Ethion	375	0.80 (1.342)	0.38 (1.174)	0.28 (1.131)	0.27 (1.127)	1.08 (1.442)	0.63 (1.278	0.47 (1.212)	0.24 (1.114)	0.29 (1.136)	0.31 (1.178)
Oxy demeton methyl	500	0.71 (1.308)	0.29 (1.135)	0.27 (1.127)	0.18 (1.086)	0.91 (1.382)	0.32 (1.148)	0.17 (1.082)	0.07 (1.034)	0.07 (1.034)	0.18 (1.086)
Untreated Control	Water spray	1.93 (1.712)	<b>2.16</b> (1.777)	2.48 (1.863)	2.33 (1.825)	3.46 (2.111)	1.64 (1.625)	1.81 (1.676)	1.897 (1.702)	2.11 (1.764)	1.15 (1.466)
CD(0.05)	· · · · · · · · · · · · · · · · · · ·	(0.1277)	(0.0682)	(0.0714)	(0.0924)	(0.1437)	(0.0875)	(0.0560)	(0.0723)	(0.0813)	(0.1005

Table 8. Mean population of mites and thrips in insecticide treated chilli plants at different intervals after first spraying at 30 days after transplanting.

Figures in the parentheses are  $\sqrt{x+1}$  values, , DAS - Days after spraying.

of the treatments recorded mite population ranging from 0.22 to 0.27/ leaf, where as untreated plants recorded 2.33 mites per leaf.

The chilli mite population increased in all the insecticidal treatments at the fourteenth day after spraying, but all the treatments were statistically superior over the untreated control (3.46 / leaf). After two weeks also the chilli mite population was significantly less in propargite (0.38 / leaf), spiromesifen (0.37 / leaf) and acetamiprid treated plants (0.56 mites per leaf) and was on par. Remaining treatments recorded average mite count ranging from 0.82 to 1.08 mites per leaf while untreated plants had a mean population of 3.46 mites per leaf.

## 4.5.1.2 Chilli thrips (S. dorsalis)

Perusal of the data (Table 8) on thrips population after one day from spraying revealed that the pest population was significantly lower in acetamiprid (0.27 / leaf), spiromesifen (0.3 / leaf), thiamethoxam (0.3 / leaf), oxy demeton methyl (0.32 / leaf) imidacloprid (0.34 / leaf) and dimethoate (0.42 / leaf). All the above treatments were on par with each other. Thrips population was comparatively high in ethion (0.63 / leaf) and propargite (0.71) but was significantly lower than in the untreated plants (1.64 / leaf).

At 3 DAS, the thrips population was statistically lower in acetamiprid treated plots (0.10 /leaf) followed by spiromesifen (0.17 / leaf), oxy demeton methyl (0.17 / leaf), imidacloprid (0.20 / leaf), and thiamethoxam (0.20 / leaf) and were on par. The rest of the treatments recorded an average thrips count ranging from 0.27 to 0.47. All the insecticidal treatments were significantly superior over the untreated plants (1.81 / leaf).

After five days, the thrips population declined rapidly in almost all the insecticidal treatments, where the population turned to zero in spiromesifen treated plots followed imidacloprid (0.02 / leaf), acetamiprid (0.02 / leaf), oxy demeton methyl (0.067 / leaf) thiamethoxam (0.09 / leaf) and dimethoate

(0.11 / leaf) were found to be equally effective in reducing thrips population and were on par with each other. Ethion (0.24 / leaf) and propargite (0.27 / leaf) recorded comparatively high population but were significantly superior over untreated control (1.897 / leaf).

At seven days after spraying, the thrips population increased slightly, but all the insecticidal treatments were significantly superior over untreated control (2.11 / leaf). Spiromesifen (0.05 / leaf), acetamiprid (0.067 / leaf), oxy demeton methyl (0.067 / leaf), thiamethoxam (0.09 / leaf), imidacloprid (0.11 / leaf) and dimethoate (0.11 / leaf) and were on par in their efficacy in controlling thrips.

At fourteen days after spraying, the thrips population increased in every insecticidal treatment. Acetamiprid (0.11 / leaf) and spiromesifen (0.13 / leaf) recorded the lowest mite population even after 14 days and were statistically on par with imidacloprid (0.22 / leaf), thiamethoxam (0.16 / leaf), dimethoate (0.26 / leaf) and oxy demeton methyl (0.18 / leaf). Ethion recorded 0.31 / leaf but was statistically superior over the untreated plants (1.15 / leaf).

# 4.5.2 Second spray at 60 DAT

#### 4.5.2.1 Chilli mite (*P. latus*)

In general, the mite population was relatively high during the period of observation taken after second spray done at 60 days after transplanting (Table 9). One day after the second spray, significantly low population of mite was observed in propargite (0.56 / leaf) and spiromesifen (0.76 / leaf) sprayed plants. All the other treatments, recorded mite population ranging from 1 to 1.4 per leaf and were significantly superior over the untreated control plants (3.33 / leaf). The mite population declined after three days of spraying. Propargite (0.24 / leaf) and spiromesifen (0.29 / leaf) recorded the lowest mite population and significantly superior over all the other insecticidal treatments.

Treatment	Dosage	Mear	population of	mites / leaf		Mean population of thrips /leaf					
. <u> </u>	g a.i.ha <sup>-1</sup>	1 DAS	3 DAS	5 DAS	7 DAS	14DAS	1DAS	3 DAS	5DAS	7DAS	14DAS
Spiromesifen	100	0.76 (1.326)	0.29 (1.135)	0.11 (1.053)	0.04 (1.021)	0.44 (1.201)	0.05 (1.023)	0.00 (1.000)	0.00 (1.000)	0.05 (1.023)	0.13 (1.064)
Imidacloprid	20	1.24 (1.497)	0.80 (1.340)	0.44 (1.201)	0.31 (1.145)	0.87 (1.365)	0.09 (1.044)	0.02 (1.011)	0.02 (1.011)	0.11 (1.052)	0.22 (1.103)
Thiamethoxam	40	1.31 (1.519)	1.04 (1.429)	0.40 (1.183)	0.36 (1.164)	1.00 (1.414)	0.16 (1.074)	0.09 (1.043)	0.09 (1.043)	0.09 (1.043)	0.18 (1.086)
Acetamiprid	20	1.00 (1.414)	0.58 (1.254)	0.13 (1.064)	0.09 (1.042)	0.58 (1.25 <b>5</b> )	0.11 (1.054)	0.00 (1.0)	0.02 (1.011)	0.07 (1.034)	0.11 (1.052)
Propargite	570	0.56 (1.249)	0.24 (1.114)	0.133 (1.063)	0.13 (1.063)	0.40 (1.183)	0.22 (1.104)	0.09 (1.043)	0.16 (1.072)	0.29 (1.134)	0.42 (1.192)
Dimethoate	300	1.4 (1.549)	1.06 (1.435)	0.60 (1.264)	0.44 (1.200)	0.93 (1.39)	0.07 (1.032)	0.04 (1.021)	0,11 (1.053)	0.11 (1.053)	0.26 (1.123)
Ethion	375	1.33 (1.527)	0.86 (1.364)	0.44 (1.201)	0.33 (1.153)	0.85 (1.358)	0.13 (1.062)	0.18 (1.086)	0.18 (1.086)	0.24 (1.114)	0.31 (1.178)
Oxy demeton methyl	500	1.29 (1.512)	1.01 (1.416)	0.49 (1.219)	0.31 (1.143)	0.81 (1.346)	0.07 (1.034)	0.04 (1.021)	0.07 (1.034)	0.04 (1.021)	0.27 (1.118)
Untreated Control	Water spray	3.33 (2.080)	3.72 (2.171)	3.81 (2.194)	3.95 (2.220)	4.17 (2.272)	0.82 (1.349)	0.91 (1.382)	1.18 (1.477)	1.11 (1.452)	1.15 (1.466)
CD(0.05)		(0.1023)	(0.0818)	(0.1109)	(0.0717)	(0.0606)	(0.0618)	(0.0802)	(0.0964)	(0.0963)	(0.1302)

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Table .9 Mean population of chilli mites and thrips in insecticide treated plants at different intervals after second spraying at 60 days after transplanting.

Figures in the parentheses are  $\sqrt{x+1}$  values, DAS - Days after spraying.

Acetamiprid (0.58 / leaf) was found as the next best treatment in reducing mite population. All the other treatments recorded mite population ranging from 0.80 to 1.06 per leaf and were stastically superior over untreated plants (3.72 / leaf).

The mite population declined again at the fifth day after spraying when spiromesifen (0.11 / leaf), propargite (0.13 / leaf) and acetamiprid (0.13 / leaf) recorded lowest mite population and were on par with each other. Thiamethoxam (0.4 / leaf), imidacloprid (0.44 / leaf), ethion (0.44 / leaf) and oxy demeton methyl (0.49 / leaf) were the next best treatments and are statistically on par. Though the plots treated with dimethoate (0.60 / leaf) recorded maximum number of mites at the fifth day of spraying, it was significantly superior over untreated plants (3.81 / leaf).

At seven days after spraying, the mite population continued to be lower in spiromesifen (0.04 / leaf), acetamiprid (0.09 / leaf) and propargite (0.13 / leaf) and were significantly superior over all the other insecticidal treatments. Remaining treatments recorded a population ranging from 0.31 to 0.44 per leaf where as the untreated plot recorded up to 3.95 mites per leaf.

After fourteen days of spraying, propargite (0.4 / leaf) and spiromesifen (0.44 / leaf) and acetamiprid (0.58 / leaf) continued to be rated as best with lowest mite population. The other insecticidal treatments recorded mite population ranging from 0.81 to 1.00 per leaf. All the insecticidal treatments were significantly superior over the untreated control which recorded a population as high as 4.17 mites / leaf.

#### 4.5.2.2 Chilli thrips (S.dorsalis)

The thrips population decreased slightly during the period of observation taken during second spray (Table 9) and one day after insecticidal spray significantly lower population of thrips was recorded in spiromesifen (0.05 / leaf), dimethoate (0.07 / leaf), oxy demeton methyl (0.07 / leaf)

imidacloprid (0.09 / leaf) and acetamiprid (0.11 / leaf) and all the above treatments were on par with each other. Rest of the treatments recorded thrips population ranging from 0.13 to 0.22 per leaf and significantly lower than the control.

No thrips population was recorded in spiromesifen and acetamiprid sprayed plants at the third day of spraying. Imidacloprid (0.02 / leaf), dimethoate (0.04 / leaf), oxy demeton methyl (0.04 / leaf), thiamethoxam (0.09 / leaf) and propargite (0.09 / leaf) were the best treatments against thrips and all these treatments were on par with each other. Comparatively higher population of thrips was noticed in ethion (0.18 / leaf) but superior over untreated plants (0.91 / leaf).

At five days after spraying, the thrips population remained the same trend as in the third day where spiromesifen recorded no thrips population, statistically superior and found on par with imidacloprid (0.02 / leaf), acetamiprid (0.02 / leaf), oxy demeton methyl (0.07 / leaf) and thiamethoxam (0.09 / leaf), dimethoate (0.11 / leaf), ethion (0.18 / leaf) and propargite (0.16 / leaf). Untreated plants recorded 1.18 thrips per leaf.

On the seventh day of spraying the thrips population increased slightly but spiromesifen (0.05 / leaf), oxy demeton methyl (0.04 / leaf), acetamiprid (0.07 / leaf), thiamethoxam (0.09 / leaf), imidacloprid (0.11 / leaf) and dimethoate (0.11 / leaf) maintained the superiority in their efficacy in controlling thrips. Propargite and ethion recorded 0.29 and 0.24 thrips / leaf respectively and untreated plants recorded a population of 1.11 thrip per leaf.

On the fourteenth day of spraying the thrips increased, and statistically lower population of thrips was recorded in acetamiprid (0.11 / leaf), spiromesifen (0.13 / leaf) and thiamethoxam (0.18 / leaf), imidacloprid (0.22 / leaf), dimethoate (0.26 / leaf), ethion (0.31 / leaf) and oxy demeton methyl (0.27 / leaf). Untreated plants recorded 1.15 thrips per leaf.

#### 4.5.3 Third spray at 90 DAT

Results relating to this item are presented in Table 10

# 4.5.3.1 Chilli mite (P. latus)

During the period of third spray given at 90 days after transplanting, the mite population in general remained the same as those after the second spray. At one day after spraying, the mite population was significantly lower in spiromesifen (0.84/leaf) and propargite (0.95 / leaf) and acetamiprid (1.11 / leaf). All the other insecticidal treatments recorded mite population ranging from 1.46 to 1.71 per leaf. All the treatments were significantly superior over the untreated plants (2.73 / leaf).

At three days after spraying, the mite population declined in the same trend as in the first day after spraying, where spiromesifen (0.53 / leaf) and propargite (0.71 / leaf) recorded the lowest mite population and spiromesifen was statistically superior over all other treatments. All the other treatments were statistically superior over the untreated plants (3.0/leaf) and recorded mite population ranging from 0.86 to 2.2 per leaf.

At five days after spraying, lowest mite population was recorded in spiromesifen (0.33 / leaf), propargite (0.42 / leaf) treated plots recorded statistically lower population and were on par. All the other insecticidal treatments recorded a mean population of 0.64 to 1.18 mites per leaf and were statistically superior over untreated control (3.11 / leaf). On the seventh day, significant reduction in the population of mite was seen in all insecticidal treatments, where as the untreated chilli plants recorded maximum mite population (3.46 / leaf). Lowest mite population was recorded in spiromesifen (0.20/leaf) followed by propargite (0.24 / leaf). The other insecticidal treatments recorded mite population ranging from 0.38 to 0.80 per leaf. Two weeks after third spray, among the different treatments, spiromesifen (0.53 / leaf) recorded the lowest mite population and statistically superior to other

Treatment	Dosage	Mean	population of	mites / leaf		Mean population of thrips / leaf					
	g a.i.haʻ	1 DAS	3 DAS	5 DAS	7 DAS	14DAS	1DAS	3 DAS	5DAS	7DAS	14DAS
Spiromesifen	100	0.84 (1.354)	0.53 (1.237)	0.33 (1.152)	0.20 (1.093)	0.53 (1.237)	0.02 (1.011)	0.00 (1.000)	0.00 (1.000)	0.02 (1.011)	0.09 (1.043)
Imidacloprid	20	1.46 (1.569)	1.20 (1.481)	0.86 (1.364)	0.53 (1.276)	1.11 (1.453)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.02 (1.011)
Thiamethoxam	40	1.56 (1.598)	1.33 (1.527)	1.18 (1.474)	0.78 (1.332)	1.44 (1.562)	0.05 (1.023)	0.02 (1.011)	0.15 (1.072)	0.11 (1.054)	0.18 (1.084)
Acetamiprid	20	1.11 (1.452)	0.86 (1.364)	0.64 (1.280)	0.38 (1.173)	0.80 (1.339)	0.04 (1.021)	0.00 (1.000)	0.00 (1.000)	0.05 (1.025)	0.07 (1.032)
Propargite	570	0.95 (1.395)	0.71 (1.307)	0.42 (1.190)	0.24 (1.114)	0.78 (1.331)	0.09 (1.043)	0.04 (1.021)	0.11 (1.054)	0.13 (1.063)	0.24 (1.114)
Dimethoate	300	1.71 (1.644)	1.29 (1.511)	1.05 (1.431)	0.80 (1.340)	1.37 (1.536)	0.07 (1.032)	0.02 (1.011)	0.04 (1.021)	0.04 (1.021)	0.11 (1.053)
Ethion	375	1.6 (1.612)	1.17 (1.474)	1.15 (1.467)	0.60 <sup>-</sup> (1.262)	1.02 (1.422)	0.09 (1.042)	0.04 (1.021)	0.13 (1.063)	0.18 (1.0846)	0.29 (1.135)
Oxy demeton methyl	500	1.46 (1.568)	2.20 (1.483)	0.95 (1.396)	0.50 (1.225)	1.27 (1.507)	0.00 (1.000)	0.00 (1.000)	0.07 (1.032)	0.07 (1.034)	0.18 (1.084)
Untreated Control	Water spray	2.73 (1.931)	3.00 (2.000)	3.11 (2.106)	3.46 (2.108)	4.00 (2.24)	0.31 (1.164)	0.44 (1.247)	0.45 (1.219)	0.44 (1.652)	0.76 (1.325)
CD(0.05)		(0.1441)	(0.0804)	(0.0734)	(0.1047)	(0.0603)	(0.0485)	(0.0451)	(0.0451)	(0.0480)	(0.0734

Table 10. Mean population of chilli mites and thrips in insecticide treated plots at different intervals after third spraying at 90 days after transplanting.

Figures in the parentheses are  $\sqrt{x+1}$  values, DAS - Days after spraying.

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treatments, followed by propargite (0.78 / leaf) and acetamiprid (0.80 / leaf). Highest mite population was recorded in the untreated plants (4.0 / leaf) and all the remaining treatments (1.11 to 1.44 mite per leaf) were significantly superior over the untreated control.

#### 4.5.3.2 Chilli thrips (S. dorsalis)

The thrips population (Table 10) was very low during the period after third spray and the population did not exceed 1 thrip/leaf. After one day, no thrips were recorded in treatments *viz.*, imidacloprid and oxy demeton methyl. Spiromesifen (0.023 / leaf), acetamiprid (0.043 / leaf), thiamethoxam (0.05 / leaf), propargite (0.09 / leaf), dimethoate (0.07 / leaf) and ethion (0.09 / leaf) were the other treatments in the order of their efficacy and were on par with each other and with the above two treatments. All the insecticidal treatments were significantly superior over the untreated control (0.31 / leaf). At 3 DAS mite population was recorded only in propargite (0.04 / leaf), dimethoate (0.02/ leaf) and ethion (0.04 / leaf) where as rest of the treatments recorded no thrips. All the treatments were on par with each other and superior over the untreated control (0.44 / leaf).

On the fifth day also same trend was noticed in the population of thrips where imidacloprid, spiromesifen, acetamiprid were devoid of thrips. Oxy demeton methyl (0.07 / leaf) and dimethoate (0.04 / leaf) followed the above treatments and were on par with each other. The other treatments propargite (0.11 / leaf) and ethion (0.13/leaf) were significantly superior over untreated control (0.45 / leaf).

At seven days after spraying, the thrips population increased slightly and the treatments imidacloprid (0 / leaf), spiromesifen (0.02 / leaf), acetamiprid (0.047 / leaf), dimethoate (0.04 / leaf) were on par and statistically superior over other treatments. The untreated plants recorded 0.44 per leaf. At fourteen days after spraying, the treatments imidacloprid (0.02 / 1eaf), acetamiprid (0.07/1eaf), spiromesifen (0.09 / 1eaf) and dimethoate (0.11 / 1eaf) were significantly superior over other insecticidal treatments. The remaining treatments recorded population ranging from 0.18 to 0.29 per leaf and were superior to untreated control (0.76 / 1eaf).

## 4.5.4 Leaf Curl Index (LCI)

The result on the effectiveness of new generation insecticides on the intensity of leaf curl caused by mites and thrips is presented in Table 11.

At 50 days after transplanting (DAT), the lowest leaf curl index (LCI) was recorded in spiromesifen sprayed plants with a mean score of 0.57 and it was significantly superior over all the other insecticidal treatments. Propargite (0.90), acetamiprid (0.97) and imidacloprid (1.13), were the next best treatments and were on par with each other. Oxy demeton methyl (1.37), dimethoate (1.43), ethion (1.53) and thiamethoxam (1.57) were the insecticides ranked in the order of their effectiveness in reducing the leaf curl of chilli when compared with the highest leaf curl index in untreated plots (2.27).

At 70 DAT, the leaf curling increased in all the insecticidal treatments, but lowest leaf curling was noticed in spiromesifen (0.97), propargite (1.07)and acetamiprid (1.23) sprayed chilli plants and were statistically superior over all the other treatments. Imidacloprid (1.43) and dimethoate (1.76) were the next best treatments and were on par. All the other insecticidal treatments recorded mean leaf curl indices ranging from 1.8 to 2.23 where as the untreated plots recorded a mean LCI of 3.70.

At 100 DAT also, the leaf curling followed the same trend as in 50 and 70 DAT where spiromesifen (0.57), propargite (0.83) and acetamiprid (1.03) recorded the lowest leaf curl index and were on par with each other. The above three treatments were significantly superior over all the other

		Le	af Curl Index	(LCI)	
Treatment	Dosage (g a.i./ha)	50 DAT	70 DAT	100DAT	Average
Spiromesifen	100	0.57	0.97	0.57	0.70
Imidacloprid	20	1.13	1.43	1.47	1.34
Thiamethoxam	40	1.57	2.0	2.2	1.92
Acetamiprid	20	0.97	1.23	1.03	1.08
Propargite	570	0.90	1.07	0.83	0.93
Dimethoate	300	1.43	1.76	1.73	1.64
Ethion	375	1:53	2.23	2.1	1.95
Oxy demeton methyl	500	1.37	1.8	1.6	1.59
Untreated Control		2.27	3.70	4.9	3.62
CD(0.05)		0.241	0.331	0.463	0.725

Table 11. Leaf Curl Index in chilli at different days after transplanting (DAT).

DAT- Days after transplanting.

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treatments. The other insecticidal treatments recorded LCI ranging from 1.47 to 2.2 and all the treatments were significantly superior over the untreated plots (4.9).

When the average leaf curl indices observed at three different stages of the crop were compared, it was seen that the lowest average leaf curl index was in spiromesifen (0.70) which was on par with treatments like propargite (0.93) acetamiprid (1.08) and imidacloprid (1.34) and all the four treatments were significantly superior over other treatments. The other insecticidal treatments, *viz.*, thiamethoxam, dimethoate, ethion and oxy demeton methyl recorded an average leaf curl index 1.92, 1.64, 1.95, 1.59 respectively, while the untreated plots recorded an average LCI of 3.62, which was statistically inferior to all the insecticidal treatments.

4.6 SAFETY EVALUATION OF NEW GENERATION INSECTICIDES ON NATURAL ENEMIES IN CHILLI ECOSYSTEM.

The results of the evaluation of new generation insecticides on the safety of natural enemies of pests of chilli are presented in Table 12 to 14.

#### 4.6.1 Predatory mite (*Amblyseius* spp)

One day after spraying, no predatory mite population was recorded on imidacloprid and ethion sprayed plots (Table 12). All the other insecticidal treatments except spiromesifen recorded population ranging from 0.02 to 0.06 where as spiromesifen treated plots (0.19 / leaf) had the highest activity of predatory mites and it was statistically superior to other insecticidal treatments. Untreated plots recorded 0.41 predatory mites per leaf. On the third day after spraying, spiromesifen treated plots recorded the highest mite population (0.07 / 1 eaf) and was on par with other insecticidal treatments. All the other treatments except oxy demeton methyl (0.01) recorded zero mite population, where as the untreated plot recorded a mite population of 0.4 /

Treatment	Mean	population o	f predatory r	nite Amblys	seius sp per	leaf.
	Dosage (g a.i./ha)	1DAS	3DAS	5DAS	7DAS	14 DAS
Spiromesifen	100	0.19 (1.091)	.07 (1.034)	0.23 (1.109)	0.10 (1.048)	0.24 (1.114)
Imidacloprid	20	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.02 (1.010)
Thiamethoxam	40	0.02 (1.010)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.10 (1.048)
Acetamiprid >	20	0.06 (1.029)	0.02 (1.010)	0.00 (1.000)	0.00 (1.000)	0.02 (1.010)
Propargite	850	0.04 (1.021)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.09 (1.044)
Dimethoate	300	0.02 (1.010)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.02 (1.010)
Ethion	375	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.01 (1.005)
Oxy demeton methyl	500	0.04 (1.021)	0.01 (1.005)	0.01 (1.005)	0.00 (1.000)	0.10 (1.048)
Untreated Control		0.41 (1.187)	0.4 (1.183)	0.47 (1.212)	0.56 (1.249)	0.8 (1.341)
CD(0.05)		(0.0467)	(0.0399)	(0.0277)	(0.0286)	(0.0277)

Table 12. Mean population of predatory mite *Amblyseius* sp at different intervals after spraying

Figures in the parentheses are  $\sqrt{x+1}$  transformed values.

DAS – Days after spraying.

leaf. The same trend was followed in the fifth day, where spiromesifen (0.23 / leaf) continued to maintain statistically high population of predatory mites, while all the other insecticidal treatments were devoid of any predatory mite population. On the fifth day of spraying, the untreated plots recorded 0.47 mite / leaf. At 7DAS, no predatory mite population was recorded in insecticidal treatments except in spiromesifen which recorded a population of 0.1 / leaf whereas the control plot recorded only 0.56 mites per leaf. Predatory mite population increased slightly two weeks after spraying and the spiromesifen (0.24 / leaf) treated plot recorded maximum population. Thiamethoxam and oxy demeton methyl were next best treatments that harbor a mean population of 0.1 per leaf, whereas the other insecticidal treatments recorded a low level of mite population ranging from 0.01 to 0.09 mites per leaf. The untreated control plots (0.8 / leaf) recorded maximum population of mites.

### 4.6.2 Coccinellid beetles

Beetles were not recorded in different insecticidal treatments except in spiromesifen and propargite which recorded a population of 0.33 beetles per five plants at one day after spraying (Table 13). The untreated plots recorded the highest population (2.33 / 5plants). On the third day of spraying, acetamiprid and propargite treated plots recorded a mean population of 0.33 beetles per five chilli plants and were statistically on par with spiromesifen treated plots which recorded the highest beetle population (0.67 / 5plants). Untreated plots recorded a mean of 2.33 beetles per five plants. At 5 DAS, spiromesifen treated plots recorded an average of one beetle per five plants. Thiamethoxam, acetamiprid, propargite and ethion recorded an average population of 0.33 beetles per five plants, while no beetle population was recorded in imidacloprid, dimethoate and oxy demeton methyl treated plots. The untreated plot recorded the highest population (3.0 / 5 plants). The beetle population increased on the seventh day and the highest population was recorded in spiromesifen (1.00 / 5 plants) followed by acetamiprid (0.67 / 5

Treatment	Dosage		Mean p	opulation of	Coc <u>cinellid be</u>	etle / 5 plants		pulation of S	oiders /5 plants		
	g a.i.ha <sup>-1</sup>	1 DAS	3 DAS	5 DAS	7 DAS	14DAS	1DAS	3 DAS	5DAS	7DAS	14DAS
Spiromesifen	100	0.33 (1.154)	0.67 (1.292)	1.00 (1.414)	1.00 (1.414)	1.67 (1.634)	0.33 (1.154)	0.67 (1.292)	1.00 ' (1.414)	1.00 (1.414)	1. <b>67</b> (1.634)
Imidacloprid	20	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.33 (1.292)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.67 (1.292)
Thiamethoxam	40	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.67 (1.292)	1.67 (1.634)	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.33 (1.154)	0.67 (1.292)
Acetamiprid	20	0.00 (1.000)	0.33 (1.154)	0.33 (1.154)	0.67 (1.292)	1.00 (1.414)	0.00 (1.000)	0.00 (1.000)	0.67 (1.292)	0.67 (1.292)	1.00 (1.41 <u>4</u> )
Propargite	570	0.33 (1.154)	0.33 (1.154)	0.33 (1.154)	0.33 (1.154)	1.00 (1.414)	0.00 (1.000)	0.33 (1.154)	0.00 (1.000)	0.00 (1.00)	0.67 (1.292)
Dimethoate	300	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.00 (1.00)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)
Ethion	375	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.33 (1.154)	0.67 (1.292)	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.33 (1.154)
Oxy demeton methyl	500	0.00 (1.000)	0.00 (1.000)	0.00 (1.000)	0.67 (1.292)	1.000 (1.414)	0.00 (1.000)	0.00 · (1.000)	0.00 (1.000)	0.33 (1.154)	0.33 (1.154)
Untreated Control	Water spray	2.33 (1.826)	2.33 (1.826)	2.00 (1.732)	3.00 (2.00)	3.33 (2.082)	1.67 (1.634)	2.33 (1.824)	3.00 (2.00)	2.33 (1.824)	1.67 (1.634)
CD(0.05)		(0.2269)	(0.2672)	(0.3199)	(0.4876)	(0.4895)	(0.1380)	(0.6005)	(0.2560)	(0.2948)	(0.3800)

Table 13. Population of coccinellid beetles and spiders in chilli ecosystem at different intervals after insecticidal spraying.

Figures in the parantheses are  $\sqrt{x+1}$  transformed values, , DAS - Days after spraying.

plants), thiamethoxam (0.67 / 5 plants) and oxy demeton methyl (0.67 / 5 plants). All the other insecticidal treatments recorded an average of 0.33 beetles per leaf, where as the untreated plot recorded the highest beetle population (3.0 / 5 plants). The beetle population increased in the chilli ecosystem two weeks after spraying where spiromesifen and thiamethoxam recorded an average of 1.67 beetles per five plants. Acetamiprid, propargite and oxy demeton methyl recorded mean population of one beetle followed by ethion (0.67 / 5 plants) and imidacloprid (0.33 / 5 plants), where as no beetles were recorded on dimethoate treated plots. The untreated plot (3.33 / 5 plants) recorded the highest population of beetles.

## 4.6.3 Spiders

Spider populations were recorded only in spiromesifen (0.33 / 5 plants) (Table 13) sprayed plants after one day of spraying where as the highest population of spiders were recorded in untreated chilli plants (1.67 / 5 plants). On the third day, the plot sprayed with propargite also recorded 0.33 spiders per five plants along with spiromesifen which harbor a mean population of 0.67 / 5 plants. Spider populations were not found in other insecticidal treatments, while the unsprayed plot recorded the highest population (2.33 / 5 plants). On the fifth day of spraying, statistically significant population was noticed in spiromesifen (1.0 / 5 plants) and acetamiprid (0.67 / 5 plants)treatments. Thiamethoxam recorded a mean population of 0.33 beetles per 5 plants. The other insecticidal sprayed plots were devoid of spiders and as same as in the earlier cases, the highest population was recorded in the unsprayed plots (3 / 5 plants). On the seventh day, the spider populations slightly increased in all the insecticidal treatments except in propargite and dimethoate which recorded zero population. The population of spiders in the spiromesifen (1.00 / 5 plants) spayed plots remained the same as in the case of the fifth day and found on par with acetamiprid (0.67 / 5plants), imidacloprid (0.33 / 5plants), thiamethoxam (0.33 / 5 plants), ethion (0.33 / 5 plants) and oxy demeton methyl (0.33 / 5plants). The highest population of spiders was recorded in unsprayed plot (2.33 / 5 plants).

After two weeks of spraying, the spider population increased in all the insecticidal treated plots except in dimethoate which didn't record any population. Spiromesifen treated and control plots recorded a mean population of 1.67 spiders per five plants which were on par with acetamiprid (1.0 / 5 plants), imidaeloprid (0.67 / 5 plants), thiamethoxam (0.67 / 5 plants) and propargite (0.67 / 5 plants) treatments.

# 4.6.4 Pollinators and Neutrals

The population of pollinators/ neutrals was high in the chilli ecosystem compared to the spiders and coccinellid beetles. (Table 14). One day after the application of insecticides, neutral insect population was recorded in spiromesifen, thiamethoxam, acetamiprid and propargite treatments which recorded an average of 0.33 per five plants. Neutrals or pollinators were not recorded in other insecticidal treatments while the unsprayed plots recorded the highest population (8.0 / 5 plants). On the third day of spraying, all the treatments except dimethoate, propargite and oxy demton methyl recorded significant population of neutrals in the crop ecosystem. The highest population was recorded in the unsprayed plot (6.67 / 5 plants). On the fifth day of spraying, neutral insects were found in all the insecticidal treatments. Spiromesifen harbor a mean of 1.33 insects followed by thiamethoxam, propargite, acetamiprid and ethion which recorded an average of 0.67 insects per 5 plants. All the other insecticides recorded an average of 0.33 / 5 plants, while the untreated plot recorded the highest neutral insect count (6.33 / 5 plants). The neutral population again increased on the seventh day, but no neutral insects were recorded in dimethoate treated plots. Maximum population was recorded in spiromesifen (1.33 / 5 plants) followed by acetamiprid (1.00 / 5plants), imidacloprid (0.67 / 5 plants), propargite (0.67 / 5 plants), ethion (0.67 / 5 plants) and oxy demeton methyl (0.67 / 5 plants) and

Treatment	Dosage	Mean popul	ation of polli	nators and	neutrals per	five plants
	(g a.i./ha)	1DAS	3DAS	5DAS	7DAS	14 DAS
Spiromesifen	100	0.33 (1.154)	0.67 (1.292)	1.33 (1.526)	1.33 (1.526)	2.33 (1.825)
Imidacloprid	20	0.00 (1.000)	0.33 (1.154)	0.33 (1.154)	0.67 (1.292)	1.33 (1.526)
Thiamethoxam	40	0.33 (1.154)	0.33 (1.154)	0.67 (1.292)	0.33 (1.154)	1.00 (1.414)
Acetamiprid	20	0.33 (1.154)	0.33 (1.154)	0.67 (1.292)	1.00 (1.414)	2.00 (1.732)
Propargite	850	0.33 (1.154)	0.00 (1.000)	0.67 (1.292)	0.67 (1.292)	1.67 (1.634)
Dimethoate	300	0.0 (1.000)	0.67 (1.292)	0.33 (1.154)	0.00 (1.000)	0.67 (1.154)
Ethion	375	0.0 (1.000)	0.0 (1.000)	0.67 (1.292)	0.67 (1.292)	1.33 (1.526)
Oxy demeton methyl	500	0.00 (1.000)	0.00 (1.000)	0.33 (1.154)	0.67 (1.292)	1.00 (1.414)
Untreated Control		8.0 (3.000)	6.67 (2.769)	6.33 (2.707)	7.00 (2.828)	7.00 (2.828)
CD(0.05)		(0.2760)	(0.3241)	(0.4561)	(0.4105)	(0.4583)

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Table 14. Population of pollinators and neutrals in chilli ecosystem at different intervals after insecticidal spraying.

Figures in the parentheses are  $\sqrt{x+1}$  transformed values.

DAS Days after spraying.

were at par. The untreated plots recorded the highest population (7.0 / leaf). After two weeks of spraying, significantly higher numbers of neutral insects were recorded in spiromesifen (2.33 / 5 plants) acetamiprid (2.00 / 5 plants), propargite (1.67 / 5 plants), ethion (1.33 / 5 plants), imidacloprid (1.33 / 5 plants) thiamethoxam (1.00 / 5 plants) and oxy demeton methyl (1.00 / 5 plants) and were at par. The untreated plot recorded maximum of 7.00 insects per five plants.

# 4.7 Biometric observations

The result on the biometric observations and yield are presented in Table 15.

#### 4.7.1 Plant height

At 30 days after transplanting (DAT) there was no significant difference in plant height among the different insecticidal treatments. At 60 DAT, spiromesifen (35.67), imidacloprid (32.00), acetamiprid (31.00), propargite (32.11) dimethoate (30.77) and oxy demeton methyl (32.50) were significantly superior and were at par. Thiamethoxam and ethion recorded 29.33 and 28.11 cm respectively. The plants in the untreated plot recorded an average height of 25.00cm.

At ninety days after transplanting, spiromesifen (49.2) recorded maximum plant height and was found on par with imidacloprid (45.66), thiamethoxam(41.00), acetamiprid (44.44), propargite (43.67), oxy demeton methyl(44.33), dimethoate (39.11) and thion (39.00) while the untreated plants recorded an average height of 33.7cm only.

#### 4.7.2 Number of terminal branches

At thirty days after transplanting, there was no significant difference in the number of branches among different insecticidal treatments. At 60 DAT, propargite (28.43) treated plants recorded maximum number of branches and was found on par with spiromesifen (26.54), acetamiprid

Treatments	Dosage	Plant height	(cm)		Branches			No of fruits	No of fruits		
	(g a.i./ha)	30DAT	60 DAT	90 DAT	30DAT	60DAT	90DAT	60DAT	90 DAT	120DAT	
Spiromesifen	100	27.55	35.67	49.2	19.22	26.54	38.00	28.00	38.00	19.33	
Imidacloprid	20	24.67	32	45.66	13.33	21.99	32.33	28.33	38.33	17.00	
Thiamethoxam	40	25.67	29.33	41.00	15.99	20.11	28.55	11.33	22.33	10.00	
Acetamiprid	20	23.67	31.00	44.44	19.10	23.67	32.33	30.00	30.33	14.00	
Propargite	570	27.45	32.11	43.67	18.33	28.43	34.89	25.67	26.67	11.33	
Dimethoate	300	24.55	30.77	39.11	13.99	17.89	26.35	14.00	20.00	8.67	
Ethion <sup>-</sup>	375	23.22	28.11	39.00	14.88	19.76	29.89	21.67	24.33	10.00	
Oxy demeton methyl	500	23.77	32.50	44.33	14.33	23.30	26.00	21.67	31.67	10.67	
Untreated Control	Water spray	23.00	25.00	33.70	10.64	15.33	23.12	11.33	15.00	6.33	
CD(0.05)		NS	5.959	7.136	NS	7.209	8.077	8.692	8.940	4.032	

100

Table 15. Plant height, terminal branches and number of green fruits at different intervals after transplanting

(23.67), oxy demeton methyl (23.3) imidacloprid (21.99). Thiamethoxam (20.11), ethion (19.76) and dimethoate (17.89) recorded lesser number of branches and was found on par with the untreated plants (15.33).

At ninty days after transplanting, maximum numbers of branches were found in spiromesifen (38) which was found on par with imidacloprid (32.33), acetamiprid (32.33) and propargite (34.89). Ethion (29.89), thiamethoxam (28.55) and oxy demeton methyl (26) recorded lesser number of branches and was found on par with untreated control (23.12).

#### 4.7.3 Number of green fruits

At sixty days after transplanting, maximum number of fruits was recorded in acetamiprid (30) which was followed by imidacloprid (28.33), spiromesifen (28), propargite (25.67), ethion (21.67) and oxy demeton methyl (21.67). All the above treatments were on par with each other. Dimethoate (14) and thiamethoxam (11.33) recorded less number of fruits and was found on par with untreated plants (11.33).

At ninty days after transplanting, maximium number of green fruits were recorded in imidacloprid (38.33) followed by spiromesifen (38), acetamiprid (30.33) and oxy demeton methyl (31.67) and all these treatments were on par with each other. The other insecticidal treatments recorded fruits in the range of 22.33 to 26.67. The untreated plot recorded an average of 15 fruits per plant.

Maximum numbers of fruits were recorded in spiromesifen (19.33) and imidacloprid (17) treated plots and were on par with each other and found significantly superior over all the other treatments at 120 DAT. The other insecticidal treatments recorded an average of 8.67 to 11.33 green fruits per plant, whereas the untreated plots recorded an average of 6.33 fruits /plant.

# 4.7.4 Yield and Benefit - Cost Ratio

The data on the yield of chilli are presented in Table 16. All the treatments resulted in significantly higher yield when compared to the control plot (1.9 kg). The insecticide treatments *viz*, spiromesifen (3.35 kg) and acetamiprid (3.12 kg) recorded significantly higher yield compared to other treatments. The other treatments thiamethoxam (2.98), imidacloprid (2.95), propargite (2.95) and oxy demetonmethyl (2.95) and ethion (2.7) were on par with each other. The dimethoate treatment recorded 2.45 kg of chili fruits per  $4m^2$  plot.

The data on the benefit-cost ratio revealed that spiromesifen gave Rs 1.97 in return for every one rupee invested as against control which gave only 1.15. The next best return was from acetamiprid and thiamethoxam which gave 1.9, 1.8 respectively. Benefit – Cost ratio of imidacloprid and propargite was 1.76. The conventional insecticides *viz.*, oxy demeton methyl, ethion and dimethoate gave 1.79, 1.64 and 1.48 in return for every one rupee invested.

# 4.8 DISSIPATION OF NEW GENERATION INSECTICIDES IN/ON CHILLI FRUITS

The result on the persistence of selected insecticides on chilli fruits are presented in table 17 to 31.

## 4.8.1 Spiromesifen

## 4.8.1.1 Method Validation

#### Recovery

The selected method gives good recovery of spiromesifen residues, 85 to 94 percentages. At 0.01 mg kg<sup>-1</sup> fortification levels, the mean recovery of spiromesifen on chilli fruits was found to be 85 per cent with a relative standard deviation (RSD) of 7.39 per cent (Table 17). At 0.1 mg kg<sup>-1</sup> the

Table 16. Yield and benefit: cost (B:C) ratio of chilli after insecticidal application.

Insecticide	Dosage (gha- <sup>1</sup> )	Mean fruit Yield (kg/4m <sup>2</sup> plot)	Yield (kg/ha)	Cost of cultivation (Rs ha <sup>-1</sup> )	Expenses for insecticides (Rs ha <sup>-1</sup> )	Total expenses (Rs ha <sup>-1</sup> )	Grosss income (Rs ha <sup>-1</sup> )	Net income (Rs ha <sup>-1</sup> )	B: C ratio
Spiromesifen	100	3.35	8375	61500	2115	63615	125625	62010	1.97
Imidacloprid	20	2.95	7375	61500	1300	62800	110625	47825	1.76
Thiamethoxam	40	2.98	7450	61500	640	62140	111750	49610	1.8
Acetamiprid	20	3.12	7800	61500	395	61895	117000	55105	1.9
Propargite	570	2.95	7375	61500	1350	62850	110625	47775	1.76
Dimethoate	300	2.45	6125	61500	412.5	61912.5	91875	29962.5	1.48
Ethion	375	2.7	6750	61500	410	61910	101250	39340	1.64
Oxy demeton methyl	500	2.95	7375	61500	360	61860	110625	48765	1.79
Untreated Control	Water spray	1.9	4750	61500		61500	71250	9750	1.15
CD(0.05)		0.357							

Insecticide	Dosage (gha- <sup>1</sup> )	Mean fruit Yield (kg/4m <sup>2</sup> plot)	Yield (kg/ha)	Cost of cultivation (Rs ha <sup>-1</sup> )	Expenses for insecticides (Rs ha <sup>-1</sup> )	Total expenses (Rs ha <sup>-1</sup> )	Grosss income (Rs ha <sup>-1</sup> )	Net income (Rs ha <sup>-1</sup> )	B: C ratio
Spiromesifen	100	3.35	8375	61500	2115	63615	125625	62010	1.97
Imidacloprid	20	2.95	7375	61500	1300	62800	110625	47825	1.76
Thiamethoxam	40	2.98	7450	61500	640	62140	111750	49610	1.8
Acetamiprid	20	3.12	7800	61500	395	61895	117000	55105	1.9
Propargite	570	2.95	7375 ·	61500	1350	62850	110625	47775	1.76
Dimethoate	300	2.45	6125	61500	412.5	61912.5	91875	29962.5	1.48
Ethion	375	2.7	6750	61500	410	61910	101250	39340	1.64
Oxy demeton methyl	500	2.95	7375	61500	360	61860	110625	48765	1.79
Untreated Control	Water spray	1.9	4750	61500		61500	71250	9750	1.15
CD(0.05)		0.357							<u> </u>

Table 16. Yield and benefit: cost (B:C) ratio of chilli after insecticidal application.

mean recovery percentage was high, 94 per cent with a RSD of 8.56 per cent. At the high concentration of 0.5 mg kg<sup>-1</sup>, the mean recovery percentage was 87 with a relative standard deviation of 5.01 per cent .

## **Calibration curve**

A calibration curve was prepared by plotting different concentrations (0.01 mg kg<sup>-1</sup>, 0.05 mg kg<sup>-1</sup>, 0.1 mg kg<sup>-1</sup>, 0.5 mg kg<sup>-1</sup> and 1 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.01-1 mg kg<sup>-1</sup> concentration (Fig 1).

## 4.8.1.2 Dissipation of spiromesifen residues in/on chilli fruits

Immediately after spraying (2 hours after spraying) of spiromesifen on chilli fruits, an initial deposit of 0.609 mg kg<sup>-1</sup> was observed in chilli fruits (Table 18). On the first day, the residue of spiromesifen was found to be 0.528 mg kg<sup>-1</sup>, which was 13.4 percentage lesser than the initial residue. On the third day, the residue of spiromesifen dissipated by 51.4 per cent (0.296 mg kg<sup>-1</sup>) and on the fifth day, the residue level declined to 0.169 mg kg<sup>-1</sup> registering a reduction of 72.3 per cent. On the seventh day, the residue was 0.03 mg kg<sup>-1</sup> whereby 95.1 per cent of the initial deposit got dissipated. No residue was detected above the level of quantitation (LOQ) at  $0.01\mu g g^{-1}$  from the tenth day of spraying.

# 4.8.2 Imidacloprid

#### 4.8.2.1 Method Validation

# Recovery

The method selected for the estimation of imidacloprid gave good recovery of the residues fortified to it, ranging from 88 to 113 per cent. (Table 19). At 0.01 mg kg<sup>-1</sup> fortification level, the mean recovery of imidacloprid was 93.3 per cent with an RSD of 9.17 per cent. At 0.05 mg kg<sup>-1</sup>, the mean recovery was 113 per cent with a relative standard deviation of 4.05 per cent.

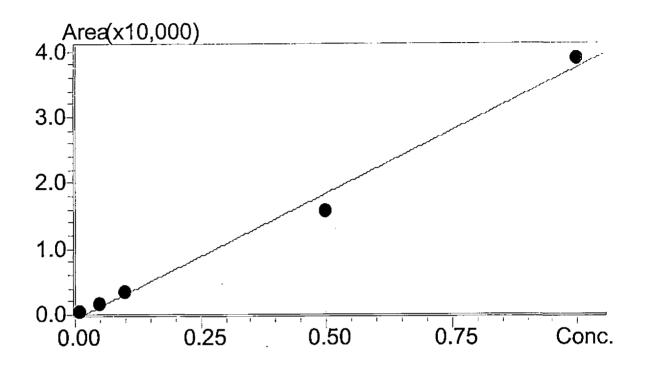
Fortification	R	covery %			
levels (mg kg <sup>-1</sup> )	R1	R2	R3	Mean recovery %	RSD (%)
0.01	79.00	84.50	91.55	85.00	7.39
0.1	102.50	86.50	93.00	94.00	8.56
0.5	84.00	85.00	92.00	87.00	5.01

Table 17. Mean recovery of spiromesifen on chilli fruits fortified at different levels.

Table 18. Residues of Spiromesifen in/on chilli fruits at intervals (days).

· · ·	Resi	due (mg kg	<sup>-1</sup> )		
Days after spraying (DAS)	R1	R2	R3	Mean $\pm$ SD	Dissipation %
0 (2 hr after spraying)	0.610	0.613	0.604	0.609±0.046	
1	0.498	0.564	0.523	0.528±0.333	13.40
3.	. 0.315	0.277	0.296	0.296±0.19	51.40
5	0.179	0.165	0.164	. 0.169±0.083	72.30
7	0.05	0.03	0.02	0.03±0.015	95.10
10	BDL	BDL	BDL	BDL	

BDL- Below detectable level, SD - Standard deviation, RSD - Relative standard deviation



Y = 38307.76X - 773.1763 R^2 = 0.9912371 R = 0.9956089

External Standard Curve: Linear Origin: Not Forced Weighting Method: None

Mean RF: 33665.1 RF SD: 2,961.725 RF %RSD: 8.797614

level	Conc.(ppm)	Mean area
1	0.01	336
2	0.05	1615
3	0.1	3216
4	0.5	15762
5	1	38798

Fig. 1. Calibration curve of Spiromesifen.

At 0.1 mg kg<sup>-1</sup> level fortification, 88 per cent of the residue was recovered with an RSD of 8.58 per cent.

## **Calibration** curve

A calibration curve was prepared by plotting different concentrations (0.05 mg kg<sup>-1</sup>, 0.1 mg kg<sup>-1</sup>, 0.2 mg kg<sup>-1</sup>, 0.3 mg kg<sup>-1</sup> and 0.4 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.05-0.4 mg kg<sup>-1</sup> concentration (Fig 2).

## 4.8.2.2 Dissipation of imidacloprid residues in/on chilli fruits

Imidacloprid applied at the rate of 50 g a.i. ha<sup>-1</sup> in chilli resulted in an initial deposit of 1.27 mg kg<sup>-1</sup> on fruits (Table 20). On the next day of spraying the residue reached 0.94 mg kg<sup>-1</sup>, resulting in 25.99 per cent dissipation. On the third day, 60.63 per cent of the residue got dissipated and recorded an average level of 0.5 mg kg<sup>-1</sup>. The residues degraded further to 81.89 per cent on the fifth day which came to mean residue level of 0.23 mg kg<sup>-1</sup>. Ninety per cent of the initial deposit got dissipated on the seventh day where the concentration was recorded to be 0.13 mg kg<sup>-1</sup>. No residue was detected thereafter from the tenth day of spraying.

## 4.8.3 Acetamiprid

#### 4.8.3.1 Method Validation

#### Recovery

The mean recovery per centage of acetamiprid when fortified at a level of 0.01 mg kg<sup>-1</sup> was 94.7 where as the relative standard deviation of 16.73 (Table 21). When fortified at 0.1 mg kg<sup>-1</sup>, the mean recovery came to around 100.16 per cent with an RSD of 20.17 per cent. Up to 95.89 per cent of the residue was recovered when fortified at a level of 0.25 mg kg<sup>-1</sup> (RSD) 22.4 per cent. When a higher concentration of 2.5 mg kg<sup>-1</sup> was fortified, the mean recovery percentage was found to be 90.06 with RSD of 1.89 per cent.

Dentification		Recovery %	6.	Maan maayami 9/	RSD (%)	
Fortification levels (mg kg <sup>-1</sup> )	R1	R2	R3	Mean recovery %	KSD (70)	
0.01	93.00	84.90	102.00	93.30	9.17	
0.05	114.00	108.00	117.00	113.00	4.05	
0.1	80.00	89.00	95.00	88.00	8.58	

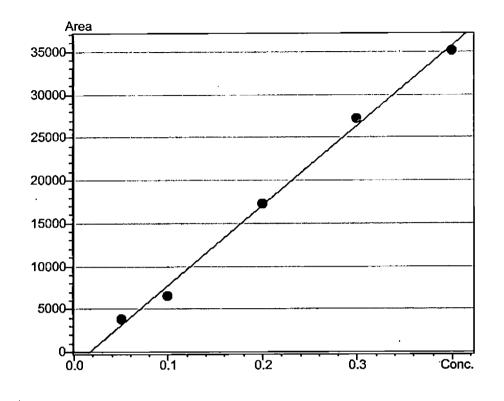
Table 19. Mean recovery of imidacloprid on chilli fruits fortified at different levels.

Table 20. Residues of imidacloprid in/on chilli fruits at intervals (days).

	R	esidue mg l	دg <sup>-1</sup>		
Days after spraying (DAS)	<b>R</b> 1	R2	R3	Mean ± SD	Dissipation %
0 (2 hr after spraying)	1.31	1.27	1.24	1.27±0.035	
1	0.93	0.88	1.01	0.94±0.065	25.99
3	0.52	0.50	0.48	0.5±0.02	60.63
5	0.25	0.21	0.23	0.23±0.02	81.89
7	0.13	0.13	0.12	0.13±0.006	89.77
10	BDL	BDL	BDL	BDL	

BDL-Below detectable level, SD - Standard deviation, RSD - Relative standard deviation

.



Y = aX + b a = 93040.26 b = -1544.307  $R^{2} = 0.9960693$ R = 0.9980327

External Standard Curve Fit Type:Linear Origin:Not Forced Weight:None

Mean RF: 81180.70 RF SD: 10434.92 RF %RSD: 12.85394

Level	Conc.(ppm)	Area
1	0.05	3756
2	0.1	6577
3	0.2	17291
4	0.3	27185
5	0.4	35160

Fig . 2. Calibration curve of imidacloprid

## Calibration curve

A calibration curve was prepared by plotting concentration vs. peak area. Good linearity was found within the range of  $0.4 - 1.0 \text{ mg kg}^{-1}$  concentration (Fig 3).

#### 4.8.3.2 Dissipation of acetamiprid residues in/on chilli fruits

Chilli fruits recorded an initial deposit of 2.44 mg kg<sup>-1</sup> (Table 22) following the application of acetamiprid @ 20 g a.i. ha<sup>-1</sup>. One day after spraying, 48.78 per cent of the initial residues dissipated reaching the level of 1.25 mg kg<sup>-1</sup>. On the third day after spraying, the plots recorded a mean residue level of 1.08 mg kg<sup>-1</sup> (dissipation of 55.74 %). On fifth day, 71.73 per cent of the residue got dissipated leaving a residue level of 0.69 mg kg<sup>-1</sup>. The level of insecticide on chilli fruits reduced to 0.47 mg kg<sup>-1</sup> on the seventh day recording a dissipation percentage of 80.74. The residues got reduced to 0.23 mg kg<sup>-1</sup> on the tenth day by dissipating 90.58 per cent from the initial load of residues. A mean residue level of 0 .015 mg kg<sup>-1</sup> was detected on the fifteenth day by degrading 99.39 per cent of the initial residue. The residue of acetamiprid was not detected on chilli fruits on the twentieth day of spraying.

## 4.8.4 Propargite

# 4.8.4.1 Method Validation

## Recovery

The method selected for the estimation of propargite gave good recovery of the residues fortified in chilli fruits, which ranged from 85.67 to 91.67 per cent (Table 23). At 0.01 mg kg<sup>-1</sup> fortification level, the mean recovery of propargite was 91.33 per cent with RSD of 5.61 per cent and at 0.05 mg kg<sup>-1</sup>; the mean recovery was 85.67 per cent with a relative standard deviation of 11.41 per cent. At 0.1 mg kg<sup>-1</sup> level fortification, 91.67 per cent of the residue was recovered with a RSD of 1.66 per cent.

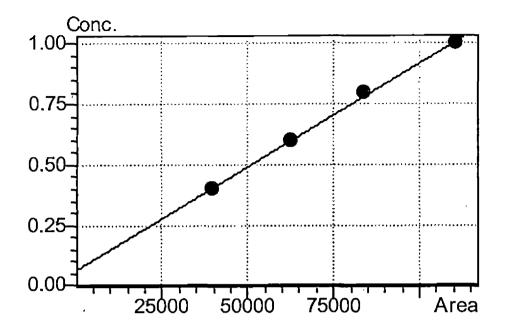
Fortification levels (mg kg <sup>-1</sup> )	Re	ecovery %	)		
	R1	R2	R3	Mean recovery %	RSD (%)
0.01	114.00	83.36	87.00	94.70	16.73
0.1	88.00	123.50	89.00	100.16	20.17
0.25	120.36	79.90	87.40	95.89	22.44
2.5	88.10	90.90	91.20	90.06	1.89

Table 21. Mean recovery of acetamiprid on chilli fruits fortified at different levels

Table 22. Residues of acetamiprid in/on chilli fruits at intervals (days)

	Residue (mg kg <sup>-1</sup> )				
Days after spraying	R1	R2	R3	Mean ± SD	Dissipation %
(DAS)					
0 (2 hr after spraying)				7	
	2.38	2.5	2.45	2.44±0.58	
1	1.25	1.22	1.27	1.25±0.029	48.78
3	1.10	1.05	1.08	1.08±0.027	55.74
5	0.69	0.70	0.66	0.69±0.022	71.73
7	0.48	0.48	0.45	0.47±0.014	80.74
10	0.23	0.24	0.22	0.23±0.009	90.58
15	0.016	0.016	0.015	0.015±0.008	99.39
20	BDL	BDL	BDL	BDL	

BDL - Below detectable level, SD - Standard deviation, RSD - Relative standard deviation



Y = aX + ba = 8.538819e-006 b = 6.628617e-002  $R^2 = 0.9981226$ R = 0.9990608

Level	Conc(ppm)	area
1	0.4	39535
2	0.6	62890
3	0.8	84017
4	1	110421

External Standard

Calib.Curve:Linear Origin:Not Forced Weight:None

Mean RF : 9.559073e-006 RF SD : 4.345824e-007 RF %RSD : 4.546282

Fig .3. Calibration Curve of acetamiprid

# **Calibration Curve**

A calibration curve was prepared by plotting concentrations (0.1 mg kg<sup>-1</sup>, 0.5 mg kg<sup>-1</sup>, 1.0 mg kg<sup>-1</sup> and 2.0 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.1-2.0 mg kg<sup>-1</sup> concentration (Fig 4).

# 4.8.4.2 Dissipation of propargite residues in/ on chilli fruits

On the day of spraying, the chilli fruits collected at 2 hours after spray recorded an average initial deposit of 1.54 mg kg<sup>-1</sup> following the application of propargite @ 570 g a.i.ha<sup>-1</sup>. On the next day 8.45 per cent of the residues got dissipated and the level reached 1.41 mg kg<sup>-1</sup> (Table 24). Fruits collected on the third day recorded an average residue level of 1.04 mg kg<sup>-1</sup> with dissipation percentage of 32.5. On the fifth day, 65.69 per cent of the residues got degraded and the concentration of propargite on chilli fruits became 0.53 mg kg<sup>-1</sup>. No residue was detected (LOQ-0.01 mg kg<sup>-1</sup>) seventh day of spraying.

# 4.8.5 Ethion

#### 4.8.5.1 Method Validation

#### Recovery

The recovery percentage of ethion at various fortification levels ranged from 76.4 to 94.9. When fortified at the lowest level of 0.01 mg kg<sup>-1</sup>, the mean recovery percentage was 94.9 and the relative standard deviation worked out was 3.88 (Table 25). At 0.1 mg kg<sup>-1</sup> level, an average of 76.4 per cent residue was recovered with RSD of 3.8 per cent. At 0.25 mg kg<sup>-1</sup>, the mean recovery percentage was 86.21 per cent and RSD was 1.87 per cent. At higher level of fortification, 0.5 mg kg<sup>-1</sup>, 77.25 per cent of residues were recovered and the RSD was calculated to be 7.54 per cent.

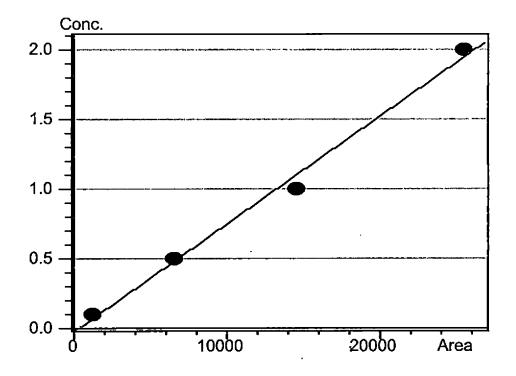
Fortification		Recovery	%	Mean % recovery	RSD (%)
levels (mg kg <sup>-1</sup> )	R1	R2	R3		
0.01	97.00	87.00	90.00	91.33	5.61
0.05	94.00	88.00	74.90	85.67	11.41
0.1	92.00	93.00	90.00	91.67	1.66
				L	

Table 23. Recovery of propargite on chilli fruits fortified at different intervals

Table 24. Residues of propargite in/on chilli fruits at intervals (days)

	Residue (mg kg <sup>-1</sup> )				
Days after spraying	<b>D</b> 1				Dissinction 9/
(DAS)	R1	R2	R3	$\underline{Mean \pm SD}$	Dissipation %
0 (2 hr after spraying)	1.53	1.53	1.55	1.54±0.012	
1	1.32	1.48	1.42	1.41±0.080	8.45
3	1.17	0.90	1.06	1.04±0.136	32.5
5	0.55	0.53	0.50	0.53±0.025	65.69
7	BDL	BDL	BDL	BDL	

BDL - Below detectable level, SD - Standard deviation, RSD - Relative standard deviation



Y= aX + b a = 7.767975e-005 b = -2.616458e-002 R^2 = 0.9927771 R = 0.9963820

External Standard Curve Fit Type:Linear Origin:Not Forced Weight:None

Mean RF : 7.610304e-005 RF SD : 4.997904e-006 RF %RSD : 6.567286 LevelConc.(ppm)Area10.1125220.5648731.01453542.025418

Fig .4. Calibration curve of propargite

# **Calibration curve**

A calibration curve was prepared by plotting different concentration  $(0.01 \text{ mg kg}^{-1}, 0.1 \text{ mg kg}^{-1}, 0.5 \text{ mg kg}^{-1} \text{ and } 1.0 \text{ mg kg}^{-1})$  vs. peak area. Good linearity was found within the range of 0.01-1.0 mg kg<sup>-1</sup> concentration (Fig 5).

## 4.8.5.2 Dissipation of ethion residues in/ on chilli fruits

The mean initial deposit of ethion on chilli fruits following the application at the rate of 375 g a.i.ha<sup>-1</sup> was found to be 5.79 mg kg<sup>-1</sup> (Table 26), which got dissipated to 3.4 mg kg<sup>-1</sup> on the first day of spraying with dissipation percentage of 41.3. On the third day, 75.83 per cent of the initial residue got dissipated and the average residue level became 1.4 mg kg<sup>-1</sup>. The dissipation continued at a slower pace and on the fifth day, the residue reduced to 1.3 mg kg<sup>-1</sup> with an average dissipation percentage of 77.55. On the seventh day, 81.52 per cent of the residue got dissipated which recorded an average level of 1.07 mg kg<sup>-1</sup>. On the tenth day, 87.92 per cent of the residue got dissipated and reached an average level of 0.7 mg kg<sup>-1</sup> on fruits. On the fifteenth day 96.72 per cent of initial residue got dissipated and recorded a mean residue of 0.19 mg kg<sup>-1</sup>. No residue was detected on the twentieth day of spraying.

#### 4.8.6 Dimethoate

## 4.8.6.1 Method Validation

## Recovery

Chilli fruits were fortified with different levels of dimethoate (0.01 mg kg<sup>-1</sup> to 1.0 mg kg<sup>-1</sup>) and the average residue recovered ranged from 73.4 to 95.7 precentage. When chilli fruits were fortified with 0.01 mg kg<sup>-1</sup> of dimethoate the mean recovery percentage was 73.4 and the relative standard deviation was 1.187 per cent (Table 27). At 0.1 mg kg<sup>-1</sup> and 0.5 mg kg<sup>-1</sup> levels, the mean recovery percentages were 95.7 and 80.3 with RSD of 13.52 and

Fortification levels (mg kg <sup>-1</sup> )	Rec	overy %		Maan 9/ maayam	
levers ( hig kg )	R1	R2	R3	Mean % recovery	RSD (%)
0.01	91.80	94.00	99.00	94.90	3.88
0.1	74.20	75.30	79.70	76.40	3.80
0.25	88.03	84.90	85.70	86.21	1.80
0.5	81.45	70.60	7 <b>9.7</b> 0	77.25	7.54

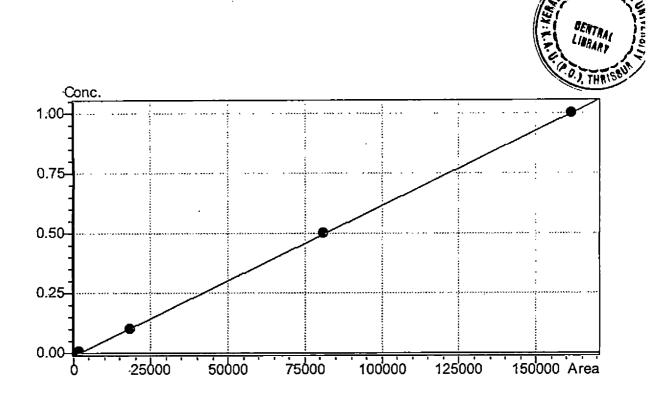
Table 25. Mean recovery of ethion on chilli fruits fortified at different levels

Table 26. residues of ethion in/on chilli fruits at intervals (days)

	Resi	idue (mg k	g <sup>-1</sup> )		
Days after spraying (DAS)	R1	R2	R3	Mean ± SD	Dissipation %
0 (2 hr after spraying)	5.96	5.67	5.73		
		·		5.79±0.153	
1	3.35	3.34	3.52	3.40±0.101	41.3
3	1.39	1.28	1.52	1.4±0.120	75.83
5	1.24	1.38	1.29	1.30±0.070	77.55
7	1.17	0.97	1.09	1.07±0.1006	81.52
10	0.80	0.59	0.71	0.70±0.105	87.92
15	0.19	0.21	0.16	0.19±0.025	96.72
20	BDL	BDL	BDL	BDL	

•

BDL - Below detectable level, SD - Standard deviation, RSD - Relative standard deviation



Y = aX + ba = 6.240772e-006 b = -7.75029e-003 R^2 = 0.9998471 R = 0.9999236

 Level
 Conc.(ppm)
 Area

 1
 0.01
 1941

 2
 0.1
 18473

 3
 0.5
 81005

 4
 1
 161529

RIGU!

External Standard Calib.Curve:Linear Origin:Not Forced Weight:None

Mean RF: 5.732108e-006 RF SD: 5.299913e-007 RF % RSD: 9.246011

Fig. 5. Calibration curve of ethion

6.82 per cent respectively. At 1 mg kg<sup>-1</sup> level of fortification, the recovery percentage was 89.16 with an RSD of 2.98 per cent.

# Calibration curve

A calibration curve was prepared by plotting concentration (0.01 mg kg<sup>-1</sup>, 0.1 mg kg<sup>-1</sup> and 1 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.01-1 mg kg<sup>-1</sup> concentration (Fig 6).

## 4.8.6.2 Dissipation of dimethoate residues in /on chilli fruits

On the day of spraying, an initial deposit of 7.63 mg kg<sup>-1</sup> (Table 28) was recorded on chilli fruits. After one day, the residue got reduced to an average of 5.42 mg kg<sup>-1</sup> with a dissipation percentage of 26.4. On the third day, the residue reduced to 37.4 % of the initial deposit and the concentration became 4.61 mg kg<sup>-1</sup>. On fifth day after spraying, the fruits recorded an average residue of 0.83 mg kg<sup>-1</sup> and the dissipation percentage increased to 88.73 per cent. On the seventh day, an average residue of 0.34 mg kg<sup>-1</sup>was recorded on chilli fruits with dissipation percentage of 95.4 per cent. On the tenth day, an average residue of 0.27 mg kg<sup>-1</sup> was recorded on chilli fruits and degradation increased slightly to 96.33 per cent. On fifteenth day 99.48 per cent residues got dissipated and an average concentration of 0.04 mg kg<sup>-1</sup> was recovered from chilli fruits. No residue was detected in chilli fruits collected on twentieth day after spraying.

#### 4.8.7 Oxy demeton Methyl

#### 4.8.7.1 Method Validation

## Recovery

The method selected for the estimation of oxy-demeton-methyl gave good recovery of the residues fortified in chilli fruits, which ranged from 76.01 to 94.66 per cent (Table 29). At 0.03 ppm fortification level, the mean recovery of propargite was 76.01 per cent with RSD of 7.38. At 0.1 mg kg<sup>-1</sup> the mean recovery was 89.66 per cent with a relative standard deviation of

SI	Fortification	Recove	ery %		Mean	RSD
No	levels (mg kg <sup>-1</sup> )	R1	R2	R3	recovery %	
1	0.01	72.40	73.80	. 74.00	73.40	1.187
2	0.1	87.90	110.67	88.60	95.70	13.52
3	0.5	75.80	78.70	86.40	80.30	6.82
4	1.0	86.40	91.70	89.40	89.16	2.98

•

Table 27. Mean recovery of dimethoate on chilli fruits fortified at different levels.

Table 28. Residues of dimethoate in/on chilli fruits at intervals (days).

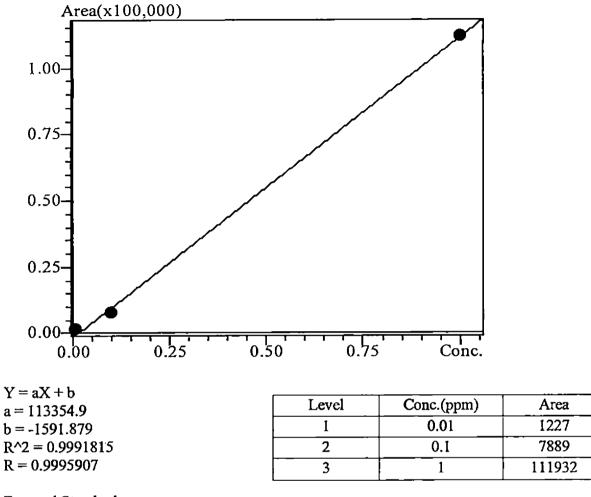
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	Res	idue (mg kg	$g^{1}$ )		
Days after spraying (DAS)	R1	R2	R3	Mean ± SD	Dissipation %
0 (2 hr after spraying)					
_	2.38	2.5	2.45	7.36±0.256	
1	1.25	1.22	1.27	5.42±0.551	26.40
3	1.10	1.05	1.08	4.61±0.101	37.40
5	0.69	0.70	0.66	0.83±0.01	88.73
7	0.48	0.48	0.45	0.34±0.01	95.40
10	0.23	0.24	0.22	0.27±0.015	96.33
15	0.016	0.016	0.015	0.04±0.006	99.48
.20	BDL	BDL	BDL	BDL	

.

BDL - Below detectable level, SD - Standard deviation, RSD - Relative standard deviation



External Standard Calib.Curve: Linear Origin: Not Forced Weight: None

Mean RF: 104523.5 RF SD: 22846.32 RF % RSD: 21.85760

Fig.6. Calibration curve of dimethoate

7.98 per cent. At 0.5 mg kg<sup>-1</sup> level 94.66 per cent of the residue was recovered with a RSD of 16.37 per cent.

## **Calibration Curve**

A calibration curve was prepared by plotting concentration  $(0.05 \text{ mg kg}^{-1}, 0.1 \text{ mg kg}^{-1}, 0.5 \text{ mg kg}^{-1}, 1 \text{ mg kg}^{-1})$  vs. peak area. Good linearity was found within the range of 0.05 - 1 mg kg<sup>-1</sup> concentration (Fig 7).

# 4.8.7.2 Dissipation of oxy demeton methyl in/on chilli fruits.

The mean residue of oxy-demeton methyl @ 500 g a.i.ha<sup>-1</sup> at different intervals is shown in table 30 which recorded a mean initial deposit of 0.966 mg kg<sup>-1</sup>, which got dissipated to 0.557 mg kg<sup>-1</sup>, with a reduction of 42.4 per cent on the first day.On the third day, the residue got reduced to 0.436 mg kg<sup>-1</sup> and the dissipation percentage increased to 54.87. The residue decline of oxy demeton methyl was at a slower pace and on the fifth day, it got reduced by 59.74 per cent and the residue level lowered to 0.389 mg kg<sup>-1</sup>. On the seventh day, the fruit sampled recorded a residue level of 0.362 mg kg<sup>-1</sup> and the degradation percentage increased to 62.6. On the tenth day, the residue decreased to an average of 0.163 mg kg<sup>-1</sup> and the dissipation percentage increased to 83.2 per cent. The residue concentration decreased to 0.049 mg kg<sup>-1</sup> on the fifteenth day, by degrading 94.3 per cent of the initial residue load, and it reached below detectable level LOQ (0.03 mg kg<sup>-1</sup>) on the fifteenth day of spraying. The residue level reached BDL on the fifteenth day of spraying.

# 4.9 HALF LIFE AND WAITING PERIOD OF DIFFERENT INSECTICIDES IN /ON CHILLI FRUITS

The half life  $(T_{1/2})$  and waiting period  $(T_{tol})$  (Table 31) of the different insecticides were calculated taking in to account the dissipation pattern and the MRL values prescribed by Central Insecticide Board and Registration Committee, Government of India and European Union. A comparison of the residue dissipation curves show that the different new generation and

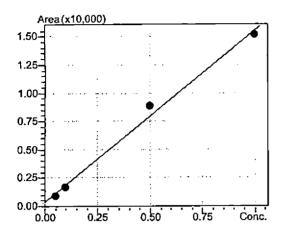
Fortification	Reco	overy %		Mean %	R <b>S</b> D (%)
levels (mg kg <sup>-1</sup> )	R1	R2	R3	recovery	
0.03	77.00	70.00	81.00 <sup>`</sup>	76.01	7.38
0.1	97.00	82.70	89.30	89.66	7.98
0.5	110.00	79.00	95.00	94.66	16.37

Table 29. Mean recovery of oxydemeton methyl on chilli fruits fortified at different interevals

Table 30. Residues oxydemeton methyl in/on chilli fruits at different intervals (days).

Days after spraying	Residue	(mg kg <sup>-1</sup> )			
(DAS)	R1	R2	R3	Mean ± SD	Dissipation %
0 (2 hr after spraying)	0.815	1.078	1.007	.966±0.136	
lodm	0.612	0.546	0.514	0.557±0.049	42.40
3	0.445	0.451	0.414	0.436±0.019	54.87
5	0.400	0.387	0.382	0.389±0.009	59.74
7	0.363	0.370	0.355	0.362±0.007	62.60
10	0.166	0.164	0.159	0.163±0.003	83.20
15	0.035	0.041	0.049	0.049±0.007	94.30
20	BDL	BDL	BDL	BDL	

BDL - Below detectable level, SD - Standard deviation, RSD - Relative standard deviation



Y = 15130.29X + 379.0054 R^2 = 0.9922661

External Standard Curve: Linear Origin: Not Forced Weighting Method: None

Level	Conc.(ppm)	Area
1	0.05	859
2	0.1	1699
3	0.5	8817
4	1	15106

Mean RF: 16727.5 RF SD: 1,114.251 RF %RSD: 6.661193 R = 0.9961255

Fig.7. Calibration curve of oxydemeton methyl

Sl No	Insecticide	MRL mg kg <sup>-1</sup>	LOQ mg kg <sup>-1</sup>	r	R <sup>2</sup>	Linear equation	T <sub>½</sub> (days)	T <sub>tol / WP</sub> (days)	
1	Spiromesifen	0.5 (EU)	0.01	-0.88	0.77	y=2.89-0.18x	1.71	7.03	
2	Acetamiprid	0.3 (EU)	0.01	-0.96	0.93	y=3.42-0.13x	2.27	3.51	
3	Imidacloprid	0.03	0.01	-0.99	0.99	y=1.62-0.17x	2.08	6.8	
4	Propargite	2(EU)	0.01	-0.96	0.94	y=2.16-0.23x	0.63	5.7	
5	Dimethoate	0.5	0.01	-0.99	0.94	y=3.9-0.15x	1.94	13.63	
6	Ethion	1	0.01	-0.97	0.98	y=3.6-0.09x	3.43	27.89	
<b>7</b>	Oxydemeton methyl	0.01(EU)	0.03	-0.97	0.95	y=2.9-0.08x	4.0	7.0	

Table 31. Half life and waiting period of different insecticides in chillies

MRL – Maximum Residue Limit

LOQ- Limit of Quantitation

r-Correlation coefficient

R<sup>2</sup>- Coefficient of determination

EU - European Union

T<sub>1/2</sub>-Half Life

T<sub>tol</sub>-Waiting period (WP)

conventional insecticides varied significantly with respect to half life and waiting period. The insecticide spiromesifen, sprayed on chilli fruits had a waiting period of 7.03 days to reach below the MRL of 0.5 mg kg<sup>-1</sup> and the time taken for half of the spiromesifen to degrade was 1.71 days. Acetamiprid had a waiting period of 3.51 days and half life of 2.27 days. Imidacloprid sprayed on chilli fruits took 2.08 days to degrade its residues to half of the initial deposit and the waiting period was fixed as 6.8 days. Propargite had a waiting period of 5.7 days and the half life was calculated as 0.63 days. Dimethoate had a long waiting period of 13.63 days on chilli fruits and a half life of 1.94 days. Ethion had the longest waiting period of 27.89 days and a half life of 4 days and a waiting period of 7 days. The LOQ for all the insecticides in the present investigation were 0.01 mg kg<sup>-1</sup> except for oxy demeton methyl which is 0.03 mg kg<sup>-1</sup>.

4.10 EFFECT OF DIFFERENT HOUSEHOLD PROCESSING TECHNIQUES IN REMOVING INSECTICIDE RESIDUES FROM CHILLI FRUITS AT 0 DAY (2 HOURS) AND 5 DAYS AFTER SPRAYING (DAS).

The effect of different house hold processing techniques in removing residues of different insecticides from chilli fruits was studied as a separate experiment and the data relating to this are presented in Table 32 and 33.

#### 4.10.1 Spiromesifen

#### 4.10.1.1 Spiromesifen (0 DAS)

At two hours after spraying of spiromesifen insecticide on chilli fruits, the unprocessed chilli fruits recorded an initial residue load of 0.65 mg kg<sup>-1</sup> (Table 32). The level of residues measured in chilli fruits when subjected to different processing techniques showed that all the decontaminating treatments significantly differed among each other. More than ninety (90.03%) per cent

of the residues were removed when the green chilli fruits were dipped in 2% tamarind solution for 20 minutes followed by washing in water and the treatment was found to be significantly superior over all the other treatments. The residue load came down to 0.066 mg kg<sup>-1</sup> by treating in tamarind solution. When the chilli fruits were dipped in 2% curd for 20 minutes and washed in water, 84.36 per cent of the initial residues were removed and residue load came down to 0.104 mg kg<sup>-1</sup>. More than eighty per cent of the residues were removed from chilli fruits, when dipped in a combination of 2% lemon + salt solution (80.96%) for 20 minutes followed by washing in water and the residue level could be reduced to 0.13 mg kg<sup>-1</sup>. About 79.41 and 75.07 per cent of the residues were removed when fruits were first dipped in alkaline medium viz, baking soda 2% and slaked lime 2% respectively followed by washing in water and the remaining residue in these cases turned out to be 0.133 and 0.17mg kg<sup>-1</sup> respectively. Dipping of spiromesifen treated chilli fruits in 2% lemon juice for 20 minutes followed by washing in water was found to remove the residues to a tune of 72.51 per cent, and the initial residual load reduced to 0.18 mg kg<sup>-1</sup>. Dipping of fruits in plain water for 20 minutes followed by washing in water could remove 66.47 per cent of the residues thereby bringing down the residue level to  $0.22 \text{ mg kg}^{-1}$ . The residue load came down to 0.23 mg kg<sup>-1</sup>, when the sprayed chilli fruits were dipped in 2% vinegar for 20 minutes followed by washing in water and by this process 65.41 per cent of the residues were removed. When the chilli fruits were subjected to dipping in 2% salt solution for 20 minutes followed by washing in plain water, 61.93 per cent of the residues were removed and the residue load came down to  $0.25 \text{ mg kg}^{-1}$ .

#### 4.10.1.2 Spiromesifen (5 DAS)

At five days after spraying, the insecticide load on unprocessed chilli fruits decreased to 0.46 mg kg<sup>-1</sup> and all the decontaminating treatments differ significantly from each other, in the extent of removal of residues (Table 33).

Treatments	pН	spiromesifen		imdacloprid		acetamiprid		propargite		ethion		dimethoate	
		Residue	%	Residue	%	Residue	%	Residue	%	Residue	%	Residue	%
		(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal
Un processed		0.65		0.6		2.59		1.95	•	4.09		7.67	
		(0.806)		(0.776)		(1.610)		(1.397)		(2.022)		(2.769)	
Water	6.15	0.22	66.47	0.06	90.05	0.06	97.69	0.32	83.71	1.78	56.48	5.15	32.86
		(0.469)		(0.235)		(0.249)		(0.596)		(1.333)		(2.268)	
Salt	5	0.25	61.93	0.04	93.37	0.23	91.15	0.4	79.64	2.12	48.17	3.63	52.67
		(0.5)		(0.210)		(0.478)		(0.631)		(1.454)		(1.904)	
Vinegar	3.14	0.23	65.41	0.05	92.33	0.15	94.23	0.23	88.29	1.16	71.63	4.85	36.77
•		(0.479)		(0.216)		(0.388)		(0.482)		(1.079)		(2.203)	
Lemon	2.32	0.18	72.51	0.06	90.05	1.48	42.86	0.25	87.27	0.69	83.13	4.05	47.20
		(0.436)		(0.249)		(1.216)		(0.501)		(0.830)		(2.012)	
Lemon + Salt	2.32	0.13	80.96	0.07	88.83	1.73	33.46	0.22	88.80	0.92	77.51	4.86	36.63
		(0.360)		(0.259)		(1.316)		(0.474)		(0.962)		(2.206)	
Curd	3.50	0.104	84.36	0.06	90.16	0.44	83.08	0.13	93.38	1.91	53.30	7.76	-1.17
		(0.322)		(0.242)		(0.662)		(0.364)		(1.381)		(2.784)	
Tamarind	3.14	0.066	90.03	0.019	96.83	2.17	16.08	0.07	96.69	1.6	60.88	3.53	53.98
		(0.257)		(0.138)		(1.473)		(0.257)		(1.266)		(1.880)	
Baking Soda	8	0.133	79.41	0.097	83.75	1.67	35.77	0.27	86.25	2.09	48.90	10.20	-32.99
		(0.364)		(0.312)		(1.294)		(0.519)		(1.449)		(3.197)	
Slaked Lime	12	0.17	75.07	0.06	90.05	0.80	69.24	0.13	93.28	2.18	46.70	7.62	0.65
		(0.412)		(0.246		(0.421)		(0.363)		(1.475)		(2.760)	
						1			_ <u></u>			<u> </u>	_1

Table 32. Extent of removal of insecticide residues from chilli fruits using different treatment solutions after 2 hours of spraying

CD (0.5) Treatment × insecticide : 0.081, Figures in the parenthesis are square root transformed values.

Maximum residue removal (77.63%) was obtained when the fruits were dipped in 2% tamarind solution followed by washing in plain water, there by bringing the residue level in the chilli fruits to  $0.10 \text{ mg kg}^{-1}$ . In the case of 2% curd treatment, 68.27 per cent of the residues could be removed, recording a residue level of 0.15 mg kg<sup>-1</sup>. When the fruits were dipped in a combination of lemon + salt (2%) solution, 62.80 per cent of the residues were removed, thereby reducing the residue level to 0.17 mg kg<sup>-1</sup>. The chilli fruits dipped in plain water for 20 minutes followed by washing with water recorded a residue load of 0.188 mg kg<sup>-1</sup>, resulting in a 58.86 per cent when compared to the unprocessed chilli fruits. When the fruits were dipped in alkaline solution of baking soda (2%) or slaked lime (2%) for 20 minutes followed by washing in water, the extent of removal was to the tune of 58.64 per cent and 57.77 per cent respectively with remaining residue levels of 0.189 and 0.19 mg kg<sup>-1</sup> respectively. By dipping in lemon solution (2%) followed by washing in water, the residue level can be brought down to 0.21 mg kg<sup>-1</sup> thereby reducing 54.92 per cent of initial residues. When chilli fruits were dipped in vinegar (2%) solution and washed with water, 50.76 per cent of the spiromesifen residues (0.23 mg kg<sup>-1</sup>) were removed. When the fruits were dipped in salt solution (2%) for 20 minutes followed by washing with plain water, only 43.10 per cent of the insecticide residues were removed and residue load was brought down to 0.26 mg kg<sup>-1</sup>.

#### 4.10.2 Imidacloprid

#### 4.10.2.1 Imidacloprid (0 DAS)

Imidacloprid residues in unprocessed chilli fruits was found to be 0.6mg kg<sup>-1</sup> at 2 hours after spraying (0 DAS). On the day of spraying all the treatment solutions were found very effective in removing the insecticide load on chilli fruits and the treatments differed significantly from each other (Table 32). About 96.83 per cent of imidacloprid residues (0.019mg kg<sup>-1</sup>) were removed when the fruits were dipped in tamarind (2%) solution for twenty

minutes followed by washing in water. Dipping the fruits 2 % salt and vinegar solution followed by washing with plain water removed residues to an extent of 93.37 per cent and 92.33 per cent respectively thereby reducing residue level to 0.04 and 0.05mg kg<sup>-1</sup> respectively. When the fruits were dipped in plain water for 20 minutes followed by washing, the residue level came down to 0.06mg kg<sup>-1</sup>, recording a residue removal of 90.05 per cent. The next best treatment was 2% curd which removed 90.16 per cent residue from fruits and the treated fruits recorded residue to the tune of 0.059mgkg<sup>-1</sup>. Chilli fruits dipped in lemon (2%) solution and a combination of lemon+salt (2%) solution followed by washing in plain water removed imidacloprid residues to the tune of 90.05 and 88.83 per cent respectively and residue load on fruits in these two treatments were 0.06 and 0.07mg kg<sup>-1</sup> respectively. The alkaline treatments viz, slaked lime (2%) and baking soda (2%) removed insecticide residues to the extent of 90.05 and 83.75 per cent and the insecticide load on chilli fruits were 0.06 and 0.097mg kg<sup>-1</sup> respectively.

#### 4.10.2.2 Imidacloprid (5 DAS)

The different decontaminating treatments were found not effective in reducing the insecticide present on the chilli fruits at five days after spraying. The unprocessed chilli fruits harvested after five days of spraying with imidacloprid recorded a low residue level of  $0.04 \text{ mg kg}^{-1}$  on fruits. (Table 33).

#### 4.10.3 Acetamiprid

#### 4.10.3.1 Acetamiprid (0 DAS)

The acetamiprid residues in the unprocessed chilli fruits was found to be 2.59 mg kg<sup>-1</sup>(Table 32) at 2 hours after spraying (0 DAS). All the decontaminating treatments were found be very effective in reducing the residue load on chilli fruits and the treatments differed significantly among each other. Dipping the fruits in plain water for 20 minutes followed by washing in plain water removed maximum amount of acetamiprid residue

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	pH	spiromesifen		imdacloprid		acetamiprid		propargite		ethion		dimethoate	
		Residue	%	Residue	%	Residue	%	Residue	%	Residue	%	Residue	%
		(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	removal	(mg/kg)	remova
Unprocessed		0.46		0.04	-	1.121		0.1		1.58		0.839	
•		(0.678)		(0.200)		(1.059)		(0.310)		(1.255)		(0.914)	İ
Water	6.15	0.19 (0.435)	58.86	0.042 (0.206)	-5.0	0.05 (0.215)	95.89	0.073 (0.270)	27	1.14 (1.069)	27.38	1.42 (1.193)	-69.04
Salt	5	0.26 (0.509)	43.10	0.047 (0.217)	-17.5	0.08 (0.292)	92.5	0.08 (0.297)	20	1.64 (1.279)	-3.79	1.14 (1.074)	-35.71
Vinegar	3.14	0.23 (0.479)	50.76	0.039 (0.194)	2.5	0.31 (0.558)	72.32	0.039 (0.198)	61	1.28 (1.135)	18.47	1.57 (1.259)	-86.90
Lemon	2.32	0.21 (0.458)	54.92	0.049 (0.221)	-22.5	0.82 (0.897)	26.9	0.047 (0.212)	53	1.04 (1.021)	33.76	2.09 (1.421)	-148.81
Lemon + Salt	2.32	0.17 (0.412)	62.80	0.042 (0.205)	-5	0.81 (0.895)	27.68	0.035 (0.187)	65	1.53 (1.236)	2.55	1.87 (1.372)	-122.62
Curd	3.50	0.15 (0.387)	68.27	0.051 (0.225)	-27.5	0.61 (0.780)	45.54	0.042 (0.207)	58	2.45 (1.561)	-56.05	1.53 (1.231)	-82.14
Tamarind	3.14	0.10 (0.319)	77.63	0.05 (0.229)	-25	0.78 (0.874)	30.5	0.065 (0.254)	35	1.28 (1.131)	18.47	1.37 (1.170)	-63.09
Baking Soda	8	0.19 (0.435)	58.64	0.06 (0.247)	-50	0.67 (0.826)	40.18	0.062 (0.249)	38	1.13 (1.063)	28.03	1.74 (1.3103)	-107.14
Slaked Lime	12	0.19 (0.435)	57.77	0.037 (0.216)	7.5	0.76 (0.868)	33.1	0.075 (0.273)	25	1.62 (1.273)	-3.18	1.71 (1.3105)	-103.57

Table 33. Extent of removal of insecticide residues from chilli fruits using different treatment solutions after 5 days of spraying

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CD (0.5) Treatment  $\times$  insecticide : 0.0589, Figures in the parenthesis are square root transformed values.

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(97.69 %) thereby bringing down the residue load to 0.06 mg kg<sup>-1</sup>. Dipping the chilli fruits in 2 % vinegar solution for 20 minutes followed by washing in water was the next effective treatment which removed 94.23% of the residue and the residue load on the fruits reduced to 0.15 mg kg<sup>-1</sup>. Dipping the chilli fruits in salt solution (2%) reduced the residue load on chilli fruits to the extent of 0.23 mg kg<sup>-1</sup> there by removing 91.15 per cent of residue. The chilli fruits dipped in 2 % solution of curd removed the residues to the extent of 83.08 per cent and the residue concentration on chilli fruits became 0.44 mg kg<sup>-1</sup>. About 69.24% of the residues were removed when the chilli fruits were dipped in alkaline medium viz, slaked lime(2%) followed by washing in plain water and the residue level on chilli fruits came down to 0.8 mg kg<sup>-1</sup>. Dipping chilli fruits in lemon (2%) solution reduced only 42.86 per cent of the residues and the residue load was reduced to 1.48 mg kg<sup>-1</sup>. The treatment of fruits by 2% baking soda and a combination of lemon + salt 2% solution followed by washing in plain water, reduced the residue level to 1.67 and 1.73 mg kg<sup>-1</sup> respectively there by removing 35.77 and 33.46 percentage of the initial residue. Dipping the chilli fruits in tamarind solution (2%) was found to be least effective in removing acetamiprid residues (16.08 %) from chilli fruits, recording a residue concentration of 2.17 mg kg<sup>-1</sup> on chilli fruits.

#### 4.10.3.2 Acetamiprid (5 DAS)

Dipping of chilli fruits in water for 20 minutes followed by washing proved to be the best treatment (95.89 % removal) in removing acetamiprid residues from chilli fruits at 5 DAS, recording a final residue concentration of 0.05mg kg<sup>-1</sup> (Table 33). The treatments by salt 2% and vinegar 2% solutions were the next best in removing residue to a tune of 92.5 and 72.32 per cent respectively resulting in a residue reduction of 0.08 mg kg<sup>-1</sup> and 0.31 mg kg<sup>-1</sup>. In all other decontamination treatments, the residue removal was below 50 per cent. Chilli fruits dipped in 2% of curd solution for 20 minutes followed by washing in water removed 45.54 of the initial residues and the residue load on the fruits came down to 0.66 mg kg<sup>-1</sup>. The alkaline treatments viz, 2 % solutions baking soda and slaked lime solution reduced the residues to 0.67 mg kg<sup>-1</sup> and 0.76 mg kg<sup>-1</sup> respectively corresponding to 40.18 per cent and 33.10 per cent of the initial residues. The fruits dipped in tamarind (2%) solution for twenty minutes followed by water wash reduced the residues to 0.78 mg kg<sup>-1</sup>, removing 30.5 per cent of the residues. When the fruits were dipped in treatments of lemon juice (2%) and a combination of lemon + salt 2% solution, the residue concentration on the fruits reduced to 0.82 and 0.81 mg kg<sup>-1</sup>, thereby removing 26.9 and 27.68 per cent respectively.

#### 4.10.4 Propargite

#### 4.10.4.1 Propargite (0DAS)

All the decontaminating treatments were found to be effective in removing the propargite residues from chilli fruits and they differed significantly among each other. Dipping of the propargite sprayed fruits in tamarind (2%) solution for 20 minutes followed by washing in water removed 96.69 per cent (Table 32) of the residues, there by bringing down the residue concentration on chilli fruits from 1.95 mg kg<sup>-1</sup> (unprocessed) to 0.065 mg kg<sup>-1</sup> <sup>1</sup>. When the fruits were dipped in treatments of curd (2 %) and slaked lime (2%) solution followed by water wash, an average of ninety three percent of the residues were removed (93.38% and 93.28%) from fruits keeping the residue concentration lower level 0.13 and 0.132 mg kg<sup>-1</sup> respectively. Treatments of fruits by vinegar, lemon and lemon+salt solution each at 2% concentration remove 88.29, 87.27 and 88.80 per cent of the residues respectively. The residues remained on chilli fruits in these treatments were 0.23, 0.25 and 0.22 mg kg<sup>-1</sup> respectively. The chilli fruits dipped in baking soda (2%) for twenty minutes followed by water wash removed 86.25 per cent of the residue and the residue concentration on the chilli fruits was brought down to 0.27 mg kg<sup>-1</sup>. The washing of fruits in water and two per cent salt solution for twenty minutes followed by washing in plain water recorded a

residue concentration of 0.32 and 0.4 mg kg<sup>-1</sup> respectively, corresponding to a removal of 83.71 and 79.64 per cent of initial residues.

#### 4.10.4.2 Propargite (5 DAS)

The decontaminating treatments were effective in removing the residues from fruits and the treatments differed significantly among each other at five days after spraving. The unprocessed fruits showed a residue concentration of 0.10mg kg<sup>-1</sup> (Table 33). Maximum residue removal of 65 per cent was recorded when the chilli fruits were dipped in a combination of lemon+salt (2%) solution and the residue load on the fruits decreased to  $0.035 \text{mg kg}^{-1}$ . Chilli fruits dipped in vinegar (2%) solution for twenty minutes followed by washing with plain water could remove 61% of the surface residues thereby reducing the residue level to 0.039 mg kg<sup>-1</sup>. About 58 per cent of the residues were removed when the fruits were dipped in 2% curd and residue level came down to 0.042 mg kg<sup>-1</sup>. When the fruits were dipped in lemon (2%) solution, 53 per cent of the residues were removed and residue concentration on the fruits reduced to 0 .047 mg/kg. Thirty eight and thirty five percentage of the residues were removed when the fruits were dipped in 2% solutions of baking soda and tamarind, recording a residue concentration of 0.062 and 0.065 mg kg<sup>-1</sup> respectively. When the fruits were dipped in plain water for 20 minutes and washed in water, only 27% of the surface residues could be removed, there by recording a residue concentration of 0.073 mg kg<sup>-1</sup>. In the alkaline medium, viz slaked lime 2% solution, about 25% of the residues were removed recording a residue concentration of 0 .075 mg kg<sup>-1</sup>. Dipping in salt solution (2%) was the least effective of the treatments removing only 20% of the initial residues resulting in a concentration of  $0.08 \text{ mg kg}^{-1}$ .

#### 4.10.5 Ethion

#### 4.10.5.1 Ethion (0DAS)

All the decontaminating treatments differed significantly in their efficacy in removing ethion residues from chilli fruits. Maximum quantity of ethion residues were removed (83.13%) when the chilli fruits were dipped in 2% solution of lemon for 20 minutes followed by washing in water and the residue concentration decreased to 0.69 mg kg<sup>-1</sup> (Table 32) from an initial level of 4.09 mg kg<sup>-1</sup> in unprocessed fruits. Chilli fruits dipped in a combination of lemon+salt (2%) solution could remove 77.51 per cent of the residues and the concentration lowered to 0.92 mg kg<sup>-1</sup> .When chilli fruits were dipped in 2% solution of vinegar, the residue concentration on the fruits became 1.16 mg kg<sup>-1</sup>, removing 71.63 per cent of the residues. More than fifty percentages of the residues were removed when the chilli fruits were dipped in treatments of 2% tamarind solution (60.88%), water (56.48%) and 2% curd (53.30 %) registering residue levels of 1.6, 1.78 and 1.91 mg kg  $^{-1}$ respectively. Chilli fruits dipped in 2% salt solution for twenty minutes followed by washing in water removed 48.17% of the initial residues and the residue concentration came down to 2.12 mg kg<sup>-1</sup>. The alkaline treatments viz baking soda (2%) and slaked lime (2%) removed 48.90 per cent and 46.70 per cent of ethion residues from chilli fruits and the residue concentration decreased to 2.09 and 2.18mg kg<sup>-1</sup> respectively.

#### 4.10.5.2 Ethion (5 DAS)

The different decontaminating treatments were found less effective in removing residues from the chilli fruits at five days after spraying. The unprocessed fruits recorded residue concentration of 1.58 mg kg<sup>-1</sup> (Table 33). Maximum amount of residues were removed (33.76%) when the chilli fruits were dipped in a 2% solution of lemon juice for twenty minutes followed by washing in water and the residue came down to 1.04 mg kg<sup>-1</sup>. Chilli fruits dipped in baking soda (2%) solution could remove only 28.03 percent of the

insecticide residues and the residue load was 1.13 mg kg<sup>-1</sup>. When the chilli fruits were dipped in plain water for twenty minutes, 27.38 per cent of the residues were removed and the residue on fruits became 1.14 mg kg<sup>-1</sup>. The residue level reduced to 1.28 mg kg<sup>-1</sup> when the chilli fruits were dipped in two per cent solutions of vinegar and tamarind there by removing 18.47 per cent of the initial residues. Fruits dipped in a combination of lemon+salt 2% solution for twenty minutes followed by washing in water could remove 2.55 per cent of residues and the residue load became 1.53mg kg<sup>-1</sup>. Fruits dipped in two per cent solutions of salt, curd and slaked lime were found not effective in removing residues.

#### 4.10.6 Dimethoate

#### 4.10.6.1 Dimethoate (0DAS)

The unprocessed chilli fruits recorded a residue concentration of 7.67mg kg<sup>-1</sup> at two hours after spraying and the decontaminating treatments differed significantly among each other. Maximum amount of residues (53.98%) were removed when the fruits were dipped in tamarind 2% solution and the residue concentration in tamarind treated chilli fruits were 3.53 mg kg<sup>-1</sup>(Table 32). The next best treatment in removing dimethoate residues (52.67%) was dipping the fruits in 2% solution of common salt and the residue load was 3.63mg kg<sup>-1</sup>. Dipping chilli fruits in lemon juice (2%) solution followed by washing removed 47.20 percent of the residues there by reducing the residue level to 4.05mg kg<sup>-1</sup>. The treatments of fruits by vinegar 2% and combination of lemon + salt 2% solutions removed 36.77 and 36.63 per cent residue respectively, reducing the residue concentration to the extent of 4.85 and 4.86 mg kg<sup>-1</sup> respectively. Dipping the chilli fruits in slaked lime 2% solution could remove only 0.65 % of the residue and the residue concentration on fruits remained to a level of 7.62 mg kg<sup>-1</sup>. The other treatments viz, curd and baking soda 2% solution did not have any effect on removing residues from the chilli fruits.

### 4.10.6.2 Dimethoate (5DAS)

The unprocessed chilli fruits recorded a residue concentration of  $0.84 \text{ mg kg}^{-1}$  at five days after spraying. None of the decontaminating treatments was found effective as evidenced by the level of residues in the various treatments. (Table 33).

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## DISCUSSION

#### 5. DISCUSSION

Monoculture of crops together with unscrupulous use of chemical insecticides and the global climate change have resulted in major pests shift from leaf or fruit eating caterpillars to sucking pests in many crop ecosystems. The major two sucking pests of chilli, thrips and mites characterised by relatively short life cycles, can complete several generations on a crop. The mechanical feeding injury and desapping by immatures and adults of these two pests on apical parts results in extensive leaf curling and subsequent reduction in yield. Several authors have reported the control options using conventional acaricides and insecticides to combat huge losses in production of chilli. According to David (1991), the application of commonly used insecticides belonging to organophosphates, carbamates and pyrethroids aimed at checking sucking pests caused resurgence of Polyphagotarsonemus latus (Banks). Besides, these chemicals are having non - target and off- target impacts on living creatures and causes ecological disturbances. Further more the rapidly developing resistance to conventional insecticides provides the impetus to develop new alternative insecticides which are broadly active on insect pests, low toxicity to vertebrates and selectivity for beneficial arthropods.

Control on pesticide residue in crops are generally based on Maximum Residue Limits (MRL) which are fixed using field trial data for a particular pesticide to arrive at the highest residue levels expected under use according to Good Agricultural Practices. Based on MRL values and the degradation pattern of insecticide residues on chilli fruits, the waiting period / pre harvest interval of different insecticides on chilli fruits can be fixed. Indiscriminate use of pesticides particularly at the fruiting stage and the non adoption of safe waiting period lead to accumulation of pesticide residues in chilli fruits. Pesticide residues in chillies have been reported by various workers in India (Nandihalli, 1979; Awasthi *et al.*, 2001; Dhotre *et al.*, 2001 and Joia *et al.*, 2001). Since food is the main source of exposure of general population to pesticides and it accounts for more than 90 per cent of total exposure (Mills., 1936) we should make sure about the safety of our daily food.

The present study provides an insight into the management of sucking pests in chilli using new generation insecticides, the safety of these insecticides to natural enemies, the dissipation pattern of different insecticides on chilli fruits and the different domestic techniques to remove the residues from the chilli fruits.

#### 5.1 MANAGEMENT OF SUCKING PEST COMPLEX IN CHILLI.

The efficacy of eight new generation insecticides *viz.*, spinosad 75 g a.i. ha<sup>-1</sup>, spiromesifen 100 g a.i. ha<sup>-1</sup>, spirotetramat 60 g a.i. ha<sup>-1</sup>, indoxacarb 60 g a.i. ha<sup>-1</sup>, imidacloprid 20 g a.i. ha<sup>-1</sup>, thiamethoxam 40 g a.i. ha<sup>-1</sup>, flubendiamide 60 g a.i. ha<sup>-1</sup> and acetamiprid 20 g a.i. ha<sup>-1</sup> was assessed in comparison with propargite 570 g a.i ha<sup>-1</sup> and dimethoate 300 g a.i ha<sup>-1</sup> as insecticide check, against three sucking pests of chilli under laboratory conditions. The three major sucking pests of chilli selected as test organisms were chilli mite *Polyphagotarsonemus latus* (Banks), chilli thrips *Scirtothrips dorsalis* Hood, and aphids *Aphis gossypii* Glover.

Laboratory evaluation of new generation insecticides against chilli mite revealed that the insecticides spiromesifen, acetamiprid and spinosad and propargite caused 100 per cent mortality of chilli mites at 48 hours after release (Fig 8) into the insecticide treated leaf disc. After 72 hours of release, dimethoate also recorded cent per cent mortality of chilli mite. There were no reports available regarding the laboratory evaluation of new generation insecticides against chilli mite. The effect of new generation insecticides on chilli thrips revealed that acetamiprid was highly toxic to chilli thrips, giving 100 per cent mortality after 24 hours (Table 4). The insecticidal treatments viz., spiromesifen, thiamethoxam, acetamiprid and dimethoate recorded cent

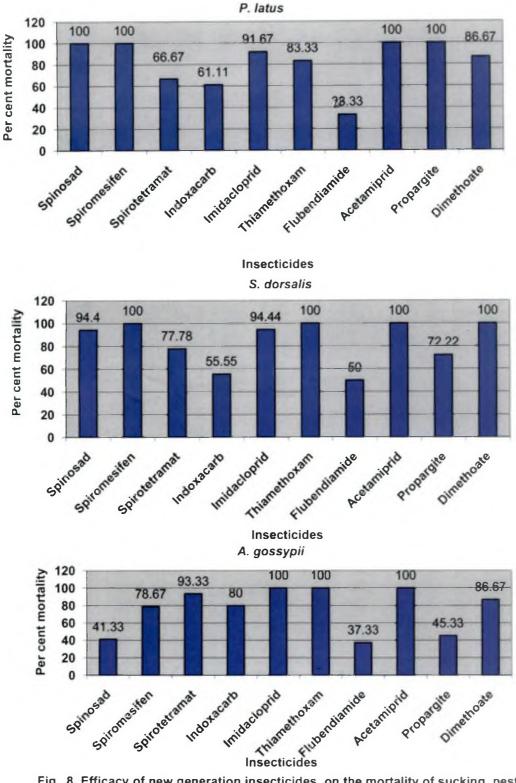


Fig. 8. Efficacy of new generation insecticides on the mortality of sucking pests of chilli exposed to insecticide treated leaf discs

per cent mortality of chilli thrips at 48 hours after release (Fig 8). The reports regarding the laboratory evaluation of new generation insecticides against chilli thrips is scanty. However Satish *et al.*, (2004) reported the superiority of acetamiprid against *Thrips palmi* (Karny) on sunflower.

The neonicotinoid insecticides, acetamiprid, thiamethoxam and imidacloprid recorded 100 per cent mortality of chilli aphids at two days after spraying (DAS) (Fig 8). At 3 DAS, 100 per cent mortality was recorded in spiromesifen, imidacloprid and dimethoate. Thamilvel, (2010) reported the effectiveness of acetamiprid 0.002 % and imidacloprid 0.003 % against chilli aphids. The toxic plant metabolites of neonicotinoid insecticides were more toxic to aphids than the parent compound (Gervais, *et al.*, 2010). When applied at field dose the neonicotinoid insecticides affect the nicotinic acetyl choline receptors and at sublethal concentrations they showed dramatic effects on the feeding behaviour of aphids, resulting in depression of honeydew excretion, wandering and subsequent death due to starvation (Nauen , 1995; Devine *et al.*, 1996)

A pot culture study was conducted to find out the effect of these new generation insecticides on chilli mites and thrips and the results are illustrated in Fig. 9. In this experiment, spiromesifen, imidacloprid, and propargite sprayed chilli plants recorded a mean population of less than one mite per leaf. In the case of chilli thrips, population was not recorded in acetamiprid sprayed chilli plants upto 7 DAS. When the damage caused due to the feeding injury of mites and thrips were recorded in terms of leaf curl index, lowest index were recorded in spiromesifen sprayed chilli plants.

Based on the above results of the laboratory and pot culture studies, the insecticides selected for the field evaluation based on their bio efficacy against *P* latus, *S* dorsalis, and *A* gossypii were spiromesifen, imidacloprid, thiamethoxam, acetamiprid, propargite and dimethoate. In addition to this, two organophosphate insecticide viz., ethion and oxy demeton methyl, which are

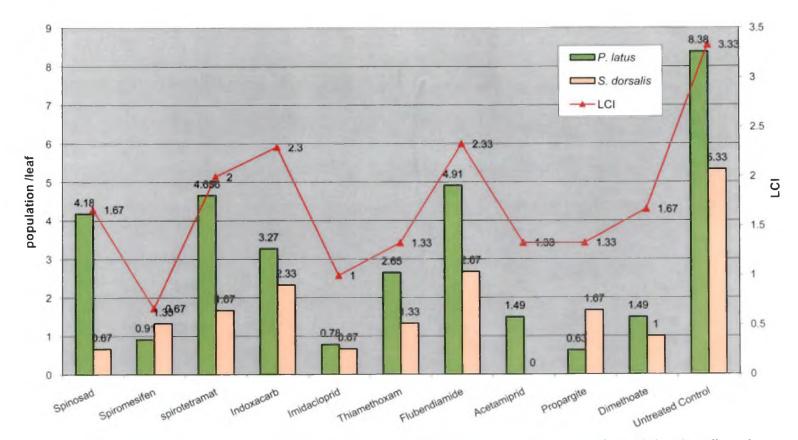
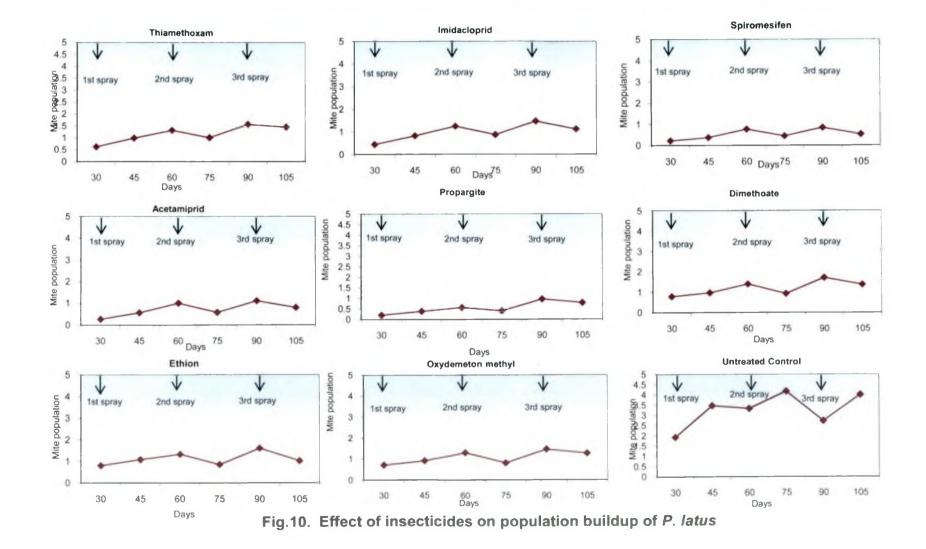


Fig. 9. Efficacy of new generation insecticides against *Polyphagotarsonemus latus*, *Scirtothrips dorsalis* and the leaf curl caused by them.

widely recommended and used for chilli cultivation were also included for subsequent evaluation. In the present investigation, chilli aphids were not a problem in the field condition, so the investigations against chilli aphids were restricted only to the laboratory conditions.

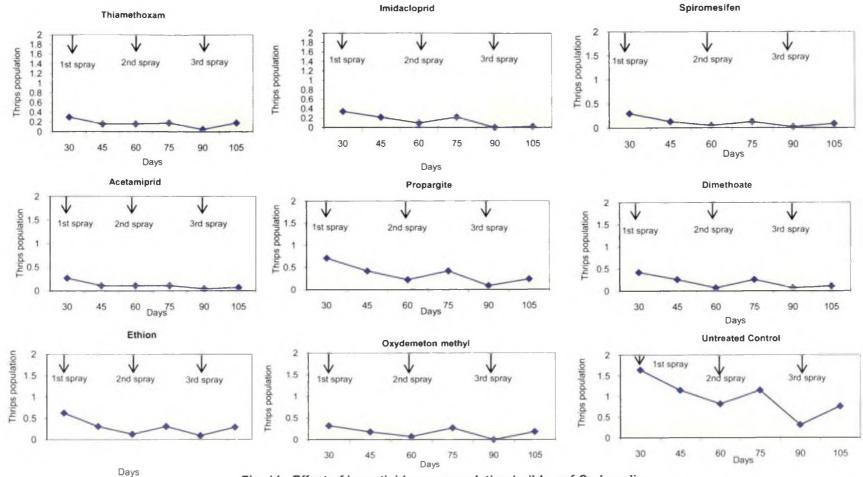
The effect of different new generation and conventional insecticides in comparison with control on the population of P. latus and S. dorsalis observed under field conditions are illustrated in Fig. 10 and Fig.11 respectively. In the field evaluation, three consecutive sprays of the selected insecticides were given at 30, 60 and 90 days after transplanting (DAT). All the insecticidal treatments were found effective in controlling mites and thrips population when compared to the untreated control. In the initial stage (vegetative) of the crop (30 DAT), the mite population was lower when compared with population of thrips. The mite population was significantly lower in propargite, spiromesifen and acetamiprid treated plots. The remaining treatments in their descending order of efficacy against chilli mite were imidacloprid, thiamethoxam, oxy demeton methyl, dimethoate and ethion. The order of efficacy of insecticidal treatments in controlling chilli thrips population were acetamiprid, thiamethoxam, spiromesifen, oxy demeton methyl, imidacloprid, dimethoate, ethion, propargite. The order of efficacies of these insecticides in controlling chilli mites and thrips remained same in the three consecutive insecticidal sprays given at 30, 60 and 90 DAT. At 90 DAT there was a sharp decline in the population of mite and thrips in the untreated plots because of the heavy rainfall prevalent during that period. The effect of the new generation and conventional insecticides on the leaf curl caused by mites and thrips were recorded at different growth stages of the plant and the results are illustrated in Fig. 12. Lowest average leaf curl index (LCI) was recorded in spiromesifen and propargite sprayed plots (Mean LCI less than 1) and were found on par with acetamiprid and imidacloprid in their efficacy in reducing leaf curl. All the insecticidal treatments were significantly superior to the untreated control. In general, the treatments given at 30, 60, 90 days after



transplanting suppressed the population of the two major sucking pests viz, thrips and mites without allowing reaching economic injury level.

It is evident from the different experiments (laboratory evaluation, pot culture study and field evaluation) that spiromesifen and propargite are very effective in reducing the mite population. Spiromesifen; a spirocyclic phenyl substituted tetronic acid derivative belongs to a new class of insecticide possessing new mode of action. It is a lipid biosynthesis inhibitor (Dekeyser, 2005). The biological activity of tetronic acids correlates with inhibition of lipogenesis especially triglycerides and free fatty acids and they are found effective against white flies and numerous mite species (Bretschneider et al, 2003). It works by preventing the treated mite or whitefly from maintaining proper water balance. This result in desiccation that is the treated mite or white fly dries up and dies. Depending upon the temperatures, the mortality of the target insects occurs with in 3 to 10 days after treatment. The superiority of spiromesifen in reducing the mite incidence was reported by several authors in chilli and other crop ecosystems. Spiromesifen had long lasting efficacy on chilli and when sprayed at 120 g a.i ha<sup>-1</sup>, it reduced the chilli mite population to 91.7 % and showed better efficacy than dicofol (Kavitha et al., 2006). The lowest mean population of chilli mite and its leaf curl index (LCI) was recorded in spiromesifen 240 SC (8.17 mites per leaf and 1.61 LCI) where as dicofol 18.5 EC, the acaricidal check recorded 13.79 mites / leaf and 2.14 LCI which differed significantly from spiromesifen (Nagaraj et al., 2007). Spiromesifen (Oberon-240SC) gave excellent control of red spider mite of brinjal along with significant increase in yield (Sekh et al., 2007).

Propargite, a new generation acaricide, which was found effective aginst chilli mite in the present investigation acts on mites by disrupting the ATP (Adenosine tri phosphate) formation by inhibiting ATP synthetase enzyme (Fishel, 2005). There are reports that propargite is a non systemic acaricide being widely used for controlling phytophagous mites infesting a





variety of crops namely cotton, vines, fruit trees and vegetables (Royal Society of Chemistry, 1987). Smitha and Giraddi, (2006a) reported that new acaricides like propargite 57 EC and fenpyroximate 5 SC recorded minimum yellow mite population in chilli and were equally effective in controlling the mites till 21 days after spray.

The superiority of neonicotinoid insecticides viz., imidacloprid, acetamiprid and thiamethoxam in reducing the chilli mite population is in line with the findings of following authors. The principal target pests of neonicotinoid insecticides are aphids, leaf hoppers, whiteflies and other sucking insects. The feeding activity of target pests ceases within minutes to hours, and death occurs usually within 24 - 48 hours but can take up to 7 days depending on the mode of application. Photostability is also an important factor in the field performance of neonicotinoids (Tomizava and Casida, 2003). Sunanda and Dethe (1998) reported that, nursery seed treatment of imidacloprid 70 WS to chilli seeds at the rate of 15g / kg seeds was very effective in keeping the chilli seedlings free from sucking pests viz. aphids, thrips and mites. In another study, it was reported that plant hole treatment (PHT) with imidacloprid (0.5g / L) effectively controlled mites and thrips (Manjunatha et al., 2000). Patil et al., (2002) reported that imidacloprid @125 ml and 150 ml/ha were highly effective against the important sucking pest complex of chilli and proved to be better than monocrotophos and dimethoate. Acetamiprid, another important member of neonicotinoid group of insecticide was reported to be very effective against aphids, leaf hoppers, whiteflies and other sucking insects and its better performance was attributed to the excellent plant mobile (systemic) property conferred by moderate water solubility (Tomizawa and Casida, 2003).

It is evident from the results that acetamiprid was very effective for controlling thrips both in laboratory and field evaluation. In addition to this, the other neonicotinoid insecticides *viz.*, imidacloprid, thiamethoxam and the tetronic acid derivative, spiromesifen were also found very effective against chilli thrips.

The effect of new generation insecticides in reducing chilli thrips was reported by several authors. Spinosad, imidacloprid and spiromesifen were found equally effective in suppressing thrips population on 'Scotch Bonnet' variety of chilli (pepper) in St.Vincent (Seal and Klassen, 2006). The superior effect of the neonicotinoid insecticides *viz*, imidacloprid, acetamiprid and thiamethoxam in reducing the chilli thrips was reported by several authors. Acetamiprid 20 SP @ 40 and 80 g a.i. /ha were effective in reducing the sucking pests of chillies followed by acetamiprid 20 SP @ 20 g a.i. /ha and recorded maximum green chilli yield (Jayewar *et al.*, 2003). The treatments with indoxacarb 14.5SC+ acetamiprid 7.7SC (RIL-042 222 SC) were significantly superior in reducing the incidence of sucking pest complex as well as fruit damage by *Helicoverpa armigera* (Hubner) (Dharne and Kabre, 2009).

Many authors (Mishra *et al.*, 2005, Sarangi and Panda, 2004, Mohan and Katiyar, 2000 and Reddy *et al.*, 2006) have reported the effectiveness of imidacloprid in reducing the thrips population in chilli. Imidacloprid was the most effective insecticide in suppressing the thrips population (average of 1.46 thrips per 10 apical leaves) and increasing the pod yield of chilli (27.63 q/ha), followed by monocrotophos and acetamiprid (Mishra *et al.*, 2005). Sarangi and Panda (2004) reported the relative field efficacy of imidacloprid, through different modes of application evaluated during 2002 and 2003 against chilli thrips. The results revealed that Seedling Root Dip (SRD) was found to be effective in reducing thrips population in chilli. Thiamethoxam, a new class of thianicotinyl compound, was found to be very effective for the control of homopterans Mohan and Katiyar (2000). Thiamethoxam 75% WS at 10 g/kg seed was significantly superior in reducing thrips up to 15days after sowing

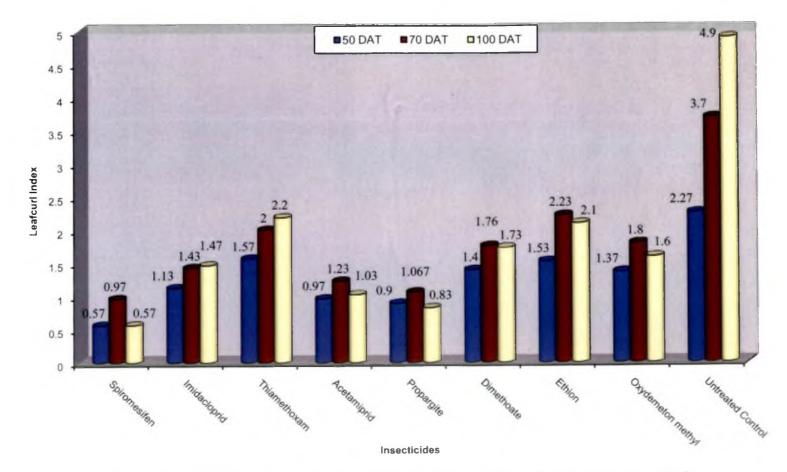


Fig. 12. Influence of new generation insecticides on the leaf curl caused both by mites and thrips

(DAS), followed by thiamethoxam 75% WS at 8 g/kg seed. (Reddy et al., 2006).

In the present study, both the conventional insecticides used in the experiment as check *viz*, oxy demeton methyl and dimethoate were found to be equally effective against thrips as new generation insecticides during the field evaluation (Fig 12). This finding is in line with findings of Mallikarjuna Rao *et al.* (1999 a) and (Soundrarajan *et al.*, 2001) where they reported that dimethoate and oxy demeton methyl were effective in reducing the thrips population in chilli plants.

The feeding of mites and thrips resulted in curling of chilli leaves and the damage was quantified by giving a score from 0 to 4, called as Leaf curl Index (LCI). Lowest leaf curl index was recorded in spiromesifen and propargite sprayed chilli plants in the pot culture study. In the field evaluation also, the same insecticides proved to be very effective in reducing the leaf curl damage. These two insecticides were found very effective in reducing mite population also when compared with other insecticides, there by minimising the leaf curl damage. So it can be concluded that the leaf curling was more due to the feeding of chilli mite than chilli thrips. The effectiveness of spiromesifen in reducing the leaf curl index is in conformity to the findings of Nagaraj *et al.*, (2007).The effectiveness of propargite in reducing the leaf curl index is in agreement with the findings of Smitha and Giraddi, (2006a).

# 5.2 SAFETY OF NEW GENERATION INSECTICIDES AGAINST NATURAL ENEMIES OF SUCKING PEST.

A laboratory experiment to assess the safety of new generation insecticides to the natural enemies of the sucking pests of chilli was conducted parallely along with the main objective and the results were illustrated in Fig. 13. The insects whose safety studied were predatory mite, *Amblyseius* spp, coccinellid grub and hemerobid grub. Analysing the data, spiromesifen can be

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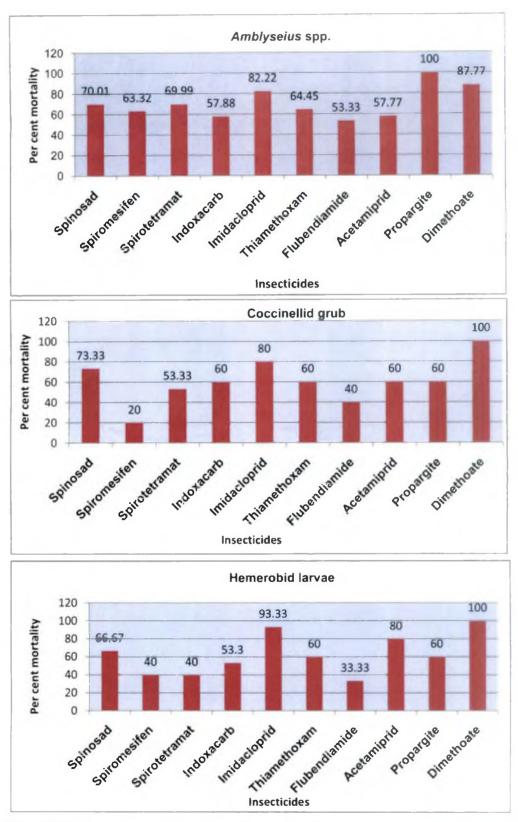


Fig. 13. Effect of new generation insecticides on natural enemies of sucking pests of chilli exposed to pesticide treated leaf discs.

handpicked in their safety to coccinellid and hemerobid grubs. Flubendiamide, spirotetramat and indoxacarb were also found moderately safe to coccinellid grubs and hemerobid larvae but were not effective for pest control. Dimethoate was found to be very toxic to coccinellid and hemerobid grubs. Among the neonicotinoid insecticides, acetamiprid and thiamethoxam were found safer than imidacloprid. In the safety evaluation against *Amblyseius* sp, acetamiprid, thiamethoxam, spiromesifen and spirotetramat were found safer as it caused less than 70 per cent mortality after 72 hours.

In the field evaluation, counts of natural enemies were taken before and after the application of insecticides which reflect the safety of different insecticides used for spraying (Table 10). The results of the field safety evaluation were illustrated in Fig. 14. In field safety evaluation against predatory mite, Amblyseius spp spiromesifen was found significantly superior in their safety against predatory mite. Propargite, dimethoate and imidacloprid treated plots were devoid of any predatory mites after application of insecticides, indicating that they are not suitable to be integrated with pest management in chilli. Similarly the counts of coccinellid beetles were not much affected by the application of spiromesifen and propargite. Acetamiprid and thiamethoxam were found moderately safer to coccinellid beetles. Spider population was not found in many of the insecticide treated plots up to 5th day after spraying. But population existed in spiromesifen and propargite treated plots and on the 14<sup>th</sup> day of spraying the spider population in the spiromesifen treated plots came on par with untreated plots. The neonicotinoid insecticides, acetamiprid and thiamethoaxam were also observed as selective insecticides when compared to imidacloprid and other conventional insecticides like ethion, dimethoate and oxy demeton methyl which harboured less number of natural enemies.

The safety of spiromesifen (Oberon-240 SC) in the present study is in agreement with the findings of Sekh and co-workers, 2007 where the

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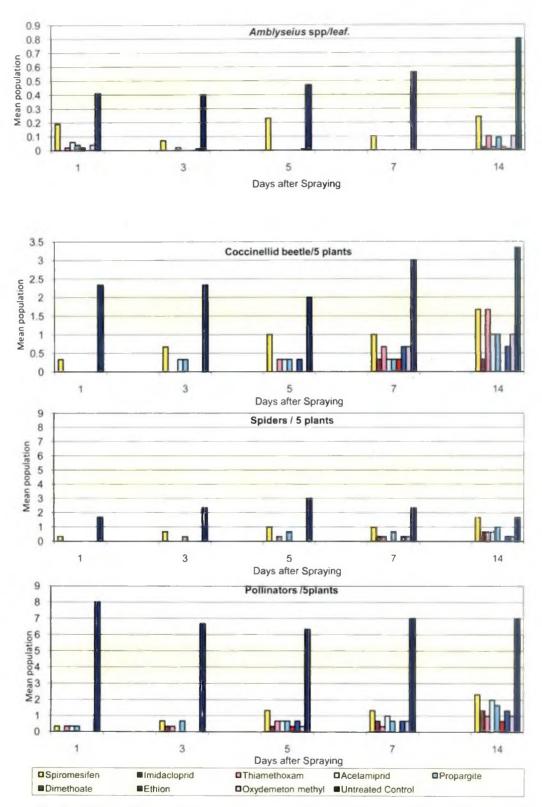


Fig. 14. Effect of insecticides on safety to natural enemies and pollinators in chilli ecosystem

chemical was found very safe to natural enemies (Stethorus sp, Chrysoperla sp, Amblyseius spp). Spiromesifen was found to be safer to Apis cerana cerana Fab and Apis mellifera L. on mustard (Choudhary et al., 2009). The safety of acetamiprid and thiamethoxam were reported previously by several authors. In experiments conducted to assess the contact toxicity to Chrysoperla carnea (Stephens) reported by Uthamasamy and co-workers (2003), less than 10% mortality was observed, irrespective of acetamiprid concentration. In a study on the toxicity of newer molecules of insecticides, acetamiprid (20 g a.i. ha<sup>-1</sup>) and thiamethoxam (25 g a.i. ha<sup>-1</sup>), were found to be relatively safer to the predatory ladybird beetles (Acharya et al., 2002). Varghese, 2003 reported that acetamiprid was safer to the aphid predator's viz., C. carnea, Menochilus sexmaculata Fabricius and Coccinella transversalis Fabricius than organophosphate insecticides like chlorpyriphos (0.05%), profenofos (0.05%) and triazophos (0.05%) on cowpea. The report of Al-Deeb et al., (2001) is in line with the present finding that thiamethoxam @ 0.002 per cent caused significantly less mortality to Orius insidiosus Say, adults than imidacloprid. Smitha and Giraddi, (2006 b) reported that propargite 57 EC and ethion 50 EC were toxic to Coccinella septempunctata L., and predatory mites, Amblyseius ovalis (Evans) and Amblyseius longispinosus (Evans), natural enemies associated with yellow mite, Polyphagotarsonemus latus in chilli which is in agreement with the present findings.

## 5.3 BIOMETRIC OBSERVATION, YIELD AND BENEFIT COST RATIO.

From the Fig 15, it is clear that the height of chilli plants in the insecticide treated plots were significantly higher to the plants in the untreated plots. Between the treatments, there is not much difference in height, however maximum height was recorded in spiromesifen treated plot which implies that spiromesifen was effective in reducing leaf curl which causes height reduction

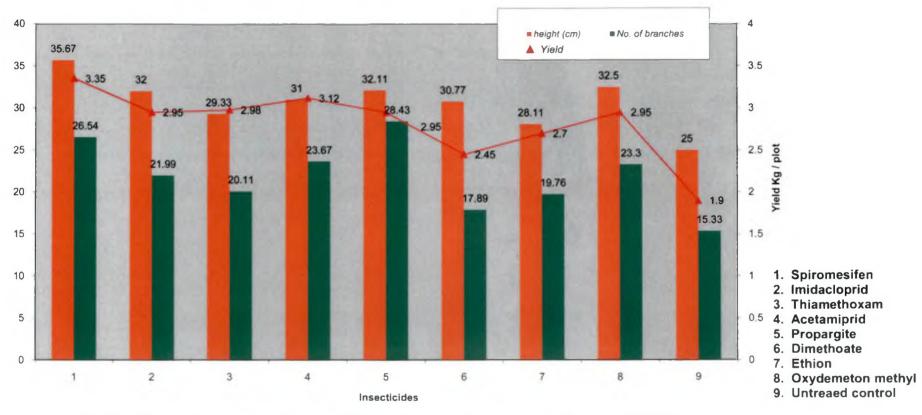


Fig. 15. Efficacy of new generation insecticides on the growth parameters and green chilli yield.

of chilli plants. Maximum number of branches were recorded in propargite treated chilli plants followed by spiromesifen and all the insecticide treated plants were statistically superior to the untreated plants (Table 15). The yield of chilli was more in spiromesifen treated plants followed by acetamiprid sprayed chilli plants.

## 5.4 PERSISTENCE AND DEGRADATION OF INSECTICIDES ON CHILLI FRUITS

As the leading producer of chilli crop in the world, India is also the largest exporter of chilli in the world. It contributes one fourth of the total quantity of chilli exported in the world followed by China which has been posing a severe competition to the Indian exporters due to variable supply and high domestic consumption. Major destinations of Indian exports are countries in West Asia, Far East, USA, Sri Lanka and Bangladesh. The exports can be further improved, provided India is able to meet the strict quality demands of international market. Recently the chilli consignments from the major chilli growing belts of Andhra Pradesh, Guntur were rejected in European countries due to the presence of residues of organophosphorus and synthetic pyrethroid insecticides. Insecticides are invariably used mainly for the control of the sucking pests, viz aphids, thrips and mites. Pesticide free products surely will boost up the export of this spice crop. Hence, it is the very need of the hour to prescribe waiting periods for the conventional and new generation insecticides used in the chilli crop.

In the present investigation, the persistence and degradation of seven insecticides used in the field evaluation were studied. The new generation insecticides whose persistence and degradation pattern studied were spiromesifen, acetamiprid and imidacloprid and the conventional insecticides whose degradation pattern studied were propargite, ethion, dimethoate and oxy demeton methyl. The dissipation pattern of the seven insecticides

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mentioned above is illustrated in Fig.16 and the degradation rate of the seven insecticides is illustrated in Fig.17.

Before sampling of chilli fruits from insecticide treated plots, recovery experiments were carried out to assess the efficiency of extraction and clean up procedures adopted and to standardize the procedure for pesticide residue estimation from chilli fruits. For validating the analytical instruments, calibration curves were plotted for each insecticide.

The selected analytical procedure gave good recovery of spiromesifen residues, 85-94 % from chilli fruits when fortified at concentration ranging from  $0.01 - 0.5 \text{ mg kg}^{-1}$ . Spraving of spiromesifen @ 100g a.i ha<sup>-1</sup> on chilli fruits deposited an initial average deposit 0. 609 mg kg<sup>-1</sup> ( Table 18 and Fig.16). More than 50 % of the residues dissipated on the third day. Sharma and co workers in 2007 reported similar results of a multilocational trial where 55% of the initial residues of spiromesifen on chilli degraded on the third day. On the seventh day about 99.6 % of the initial residues were degraded and on the tenth day the residues were below detectable level. The residues of spiromesifen on chilli fruits declined progressively with time and the half life calculated from the regression equation was found to be 1.71 days. (Table. 31). In another study the half life of spiromesifen on chilli fruits was found to be only 2 days (Sharma et al., 2007). The half life of spiromesifen in brinjal has been reported two or three days (Sharma et al., 2006b). Maximum residue limit (MRL) of spiromesifen for chilli has not been fixed in India by PFA (Prevention of Food Adulteration Act). So the MRL value prescribed by the European Union (EU) for spiromesifen (0.5 mg kg<sup>-1</sup>) is used for calculating waiting period. The waiting period of spiromesifen on chilli fruits was calculated as 7.03 days (Table 31). So in the case of spiromesifen on chilli fruits, a safety interval of minimum 8 days should be kept between last spraying and fruit harvest.

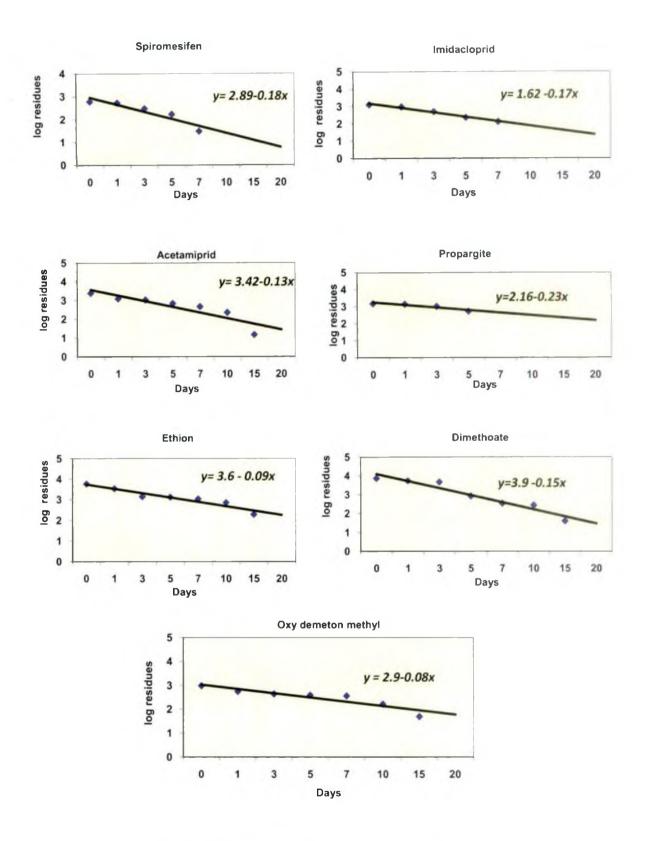


Fig. 16. Dissipation pattern of insecticides on chilli fruits

Spiromesifen did not translocate to the fruit in any of the crops like tomato, cotton, apple, brinjal and lettuce under field conditions (Weber, 2005). First metabolite of spiromesifen was formed by hydrolysis of the ester group to form enol, which was further metabolized by the hydroxylation of the methyl groups or cyclopentyl ring. The degradation rate of spiromesifen in aerobic soil has been reported in the range of 3 to 18 days, and in anaerobic conditions (water/ sediment) as 4 to 11 days, the enol and 4-carboxy spiromesifen being the major metabolites (Babezinski and Arthur, 2005).

The selected analytical procedure gave good recovery of imidacloprid residues, 88-113 per cent from chilli fruits when fortified at concentration ranging from 0.01 –0.1 mg kg<sup>-1</sup> (Table 19)<sup>-</sup> Spraying of imidacloprid 17.8 g a.i. ha<sup>-1</sup>, left an initial average residue of 1.27 mg kg<sup>-1</sup> on chilli fruits. Dikshit and Pachauri, 2000 reported that imidacloprid when applied at the rate of 20 and 40 g a.i. ha<sup>-1</sup>, gave initial deposits of 1.35 and 2.40 mg kg<sup>-1</sup> respectively on tomato fruits. More than 60 per centage of the residue got dissipated on the third day and about 90 per centage of the residue got dissipated on the seventh day of spraying (Fig. 16). The residue of imidacloprid was below detectable level on the tenth day of spraying. The dissipation data followed first order kinetics and the half life calculated from the regression equation was 2.08 days (Table 31).

Metabolites of imidacloprid detected in the eggplant, cabbage, and mustard plants included the urea derivative [1-(6-chloropyridin-3- ylmethil) imidazolidin-2-one] and 6-chloronicotinic acid at 10 days after foliar application. Foliar half lives of 3 to 5 days were calculated on these three crops when imidacloprid were applied at the rate of 20 to 40 g a.i. ha<sup>-1</sup>(Gervais et al., 2010). In green chillies, the initial residues of imidacloprid were 0.38 and 0.56 ppm in a spray treatment of 100 and 150 ml ha<sup>-1</sup> respectively and these residues reached below detectable level (BDL) of more than 0.05 ppm in 4.19 to 5.48 days (Kharbade *et al.*, 2003). Indumathi and co-workers (2001)

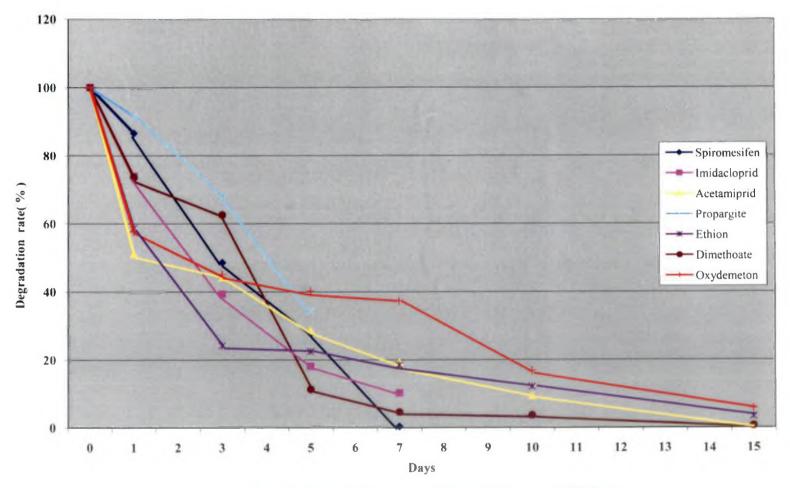


Fig.17. Degradation rate of insecticides on chilli fruits

reported that the residues of imidacloprid (Confidor 200SL) at 0.3 and 0.6 ml  $\Gamma^1$  dissipated exponentially with time following foliar application in okra with half- life of 2-4 days in two consecutive seasons which is in conformity with the present results. The residue of imidacloprid was below detectable level on the tenth day of spraying. Based on the MRL value of 0.03 mg kg<sup>-1</sup> fixed by PFA in India, a waiting period of 6.8 days was calculated for imidacloprid on chilli (Table. 31). Hence it is recommended that green chillies can be harvested 7 days after application of imidacloprid for safe consumption.

The recovery of acetamiprid for the selected analytical procedure on chilli fruits was in the range of 90.06 to 100.16 at the concentration of 0.01 to 2.5mg kg<sup>-1</sup> (Table 21). The initial residues of acetamiprid on chilli fruits at the time of spraying was at the level of 2.44 mg kg<sup>-1</sup>. More than 50 % of the residues got dissipated on the third day. The residues persisted up to fifteenth day in/on the fruits and on the fifteenth day 99.48 % of the residues got dissipated (Fig. 16). The residue of acetamiprid was below detectable level on  $20^{\text{th}}$  day of spraying. In the present study the half life (T<sub>1/2</sub>) of acetamiprid on chilli fruits calculated from the regression equation were found to be 2.27 days. When applied at the rate of 75 g a.i.ha<sup>-1</sup>on okra, the half life for acetamiprid was 2.3 days (Singh and Kulshrestha, 2005). The half life of acetamiprid 20 SP and acetamiprid 20 SL when applied at the rate of 20 g a.i.ha<sup>-1</sup> on chilli were 4.84 and 4.23 days respectively (Sanyal et al., 2008). The persistence data showed that the degradation of acetamiprid was faster in the first week, when more than 70 % of the residues got dissipated. The remaining 30 per cent of the residues got degraded in the second week of spraving. Based on the MRL of 0.3 mg kg<sup>-1</sup> fixed by European Union, a waiting period of 3.51 days is recommended for the safe consumption of acetamiprid sprayed chilli fruits. Of the various insecticides studied in the present investigation, acetamiprid is having the shortest waiting period which ensures the safety of the compound. There are reports that acetamiprid degrades rapidly and poses low risk to the environment (USEPA, 2002).

The selected analytical procedure gave good recovery of propargite residues, 91.33, 85.67 and 91.67 per cent at 0.01, 0.05 and 0.1 mg kg<sup>-1</sup> respectively (Table 23). On the day of spraying, the chilli fruits collected at 2hrs after spray recorded an average initial deposit of 1.54 mg kg<sup>+1</sup> propargite residues. In the case of propargite the dissipation pattern was different from other insecticides. About thirty per cent of the residues got dissipated quickly between third and fifth day and the residue came below detectable level from the seventh day of spraying. From the dissipation data a half -life of one day was calculated for propargite (Fig. 16). A waiting period of six days was recommended for propargite and based on the MRL values prescribed by the European Union. In another similiar field trial, the residues of propargite were estimated in brinjal fruits by High Performance Liquid chromatography (HPLC) following application of Omite 57EC @ 570 and 1140 ga.i.ha<sup>+1</sup>. The average initial deposits were 0.51 and 0.92 mg kg<sup>-1</sup> respectively which was below the maximum residue limit of  $2mg kg^{-1}$ . The half life values (T<sup>1</sup>/<sub>2</sub>) of propargite were worked out as 3.07 and 3.54 days respectively and a waiting period of one day has been suggested for safe consumption of fruits. (Kang et al., 2009).

The recovery percentage of ethion for various fortification levels at 0.01, 0.1, 0.25 and 0.5 mg kg<sup>-1</sup> were 94.9, 76.4, 86.21 and 77.25 respectively (Table 25). About seventy five per cent of the ethion residues get dissipated on the third day of spraying. After the initial fast dissipation of ethion residues the dissipation continued at slower pace up to the tenth day. On the tenth day 87.92% of the residue gets dissipated and 96.72% of the residues get dissipated on the fifteenth day (Fig.16). The residues were below detectable level on the on the twentieth day of spraying. Based on the dissipation data the half life (T<sub>1/2</sub>) values of ethion and 3.43 days .The MRL value has been fixed as 0.1 mg kg<sup>-1</sup> for ethion and based on this value, a waiting period of 28 days were calculated for ethion (Table 31). Among the different insecticide molecules studied in the present investigation ethion was having the longest

waiting period and this finding justifies the report of ethion residues in chilli consignments to European countries. There are reports that chilli consignments from India were rejected in European countries ( U.K, Italy, Greece, Germany, Spain and Netherlands ) due to the presence of aflatoxin, Escheria coli and pesticide residues of ethion, triazophos, chlorpyriphos, and dicofol (Sreenivasa cypermethrin, fenvalverate phosphamidon, Rao, 2005). In similar studies, ethion residues were below MRL value (0.5mg kg<sup>-1</sup>) after seven days of application on cucumber at the recommended dosage of 375 g a.i.ha<sup>-1</sup> (Singh et al, 2007). A waiting period of 3, 6, 10 and 15 can be suggested for different dosage levels of 500, 750, 1000 and 2000 g a.i. ha<sup>-1</sup> respectively of ethion in chilli (Joia et al., 2001). A half life of 9.4 and a waiting period (T<sub>tol</sub>) of 7.16 days were calculated for ethion in chilli (Mahalingappa et al., 2006) and the difference in waiting period with the present study may be due to the different agroclimatic conditions of the locations selected for the study.

When chilli fruits were fortified with 0.01, 0.1, 0.5 and 1.0 mg kg<sup>-1</sup> of dimethoate, the mean recovery per cent were 73.4, 95.7 80.3 and 89.16 respectively (Table 27). On the day of spraying, an initial deposit of 7.63 mg kg<sup>-1</sup> of dimethoate was recorded on chilli fruits. More than eighty per cent of the residues dissipated on the fifth day of spraying. The dissipation of dimethoate was slower during the second week of spraying that is on 7<sup>th</sup>, 10<sup>th</sup> and 15<sup>th</sup> day of spraying (Fig. 16). On the twentieth day of spraying the residue was below detectable level. From the dissipation data, the half life (T<sub>1/2</sub>) values of dimethoate in India. Based on the MRL values a waiting period of 13.63 days were calculated for dimethoate (Table 31). In a similar study, the residues of dimethoate at 0.05 and 0.1 % dissipated at the half life of 3.7 and 5.04 days and suggested a pre harvest interval (PHI) of 3 and 5 days respectively in papaya (Ahuja *et al.*, 2005). The waiting period (T tol) for dimethoate 30 EC applied @ 300 g a.i. ha<sup>-1</sup> in chillies was worked out as one

day (Reddy *et al.*, 2007b). In chilli usually plucking is done in five to six days interval and if the farmers were not observing appropriate waiting period for plucking chilli fruits, the residues of dimethoate can be problem in harvest.

The recovery per centage of oxy demeton methyl at concentrations, 0.03, 0.1 and 0.5 mg kg<sup>+1</sup> were 76.01, 89.63 and 94.68 per cent respectively (Table 29). Fifty five per cent of the oxy demeton methyl residue got dissipated on the third day of spraying. There after the residue dissipation continued at a slower pace and residues were below detectable level on fifteenth day of spraying (Fig. 16). Based on the dissipation data a half life of 4 days was calculated for oxy demeton methyl. There are no reports regarding the dissipation of oxy demeton methyl residues in vegetables. For oxydemeton methyl, based on the MRL value (0.01 mg kg<sup>-1</sup>) fixed by the European Union (EU), a waiting period of seven days has been calculated in chilli.

# 5.5 EXTENT OF REMOVAL OF INSECTICIDE RESIDUES FROM CHILLI FRUITS BY COMMON PRACTICES.

In this experiment, the effects of different processing techniques in removing the insecticide residues on chilli fruits harvested at two hours after spraying and at five days after spraying were studied. The removal of residues was also expressed in terms of processing factor which is the concentration of pesticide residue in the processed sample divided by the concentration of the residue in the unprocessed sample. Processing factors that are less than one (<1) indicate reduction of pesticide residue while those greater than one (>1) indicate concentration of pesticide residue. The different treatments include washing the chilli fruits in two per cent solutions of salt, vinegar, lemon, lemon + salt, curd, tamarind, baking soda and slaked lime, followed by washing in plain water. The insecticides selected for the present investigation were spiromesifen, imidacloprid, acetamiprid, propargite, ethion and

dimethoate. Insecticide wise and treatment wise comparison of processing factors is illustrated in Fig. 18 to 21. For the ease of discussing the results obtained in the present investigations, the percentage removal of the insecticide residues in each treatment (based on Table. 32 and Table. 33) were taken in to account rather than the processing factors.

At two hours after spraying, when the insecticide applied chilli fruits were dipped in water for 20 minutes followed by washing in tap water, maximum residues got removed in the case of acetamiprid sprayed chilli fruits (97.69%) followed by imidacloprid (90.05), propargite (83.71), spiromesifen (66.47), ethion (56.48%) and dimethoate (32.86%) (Table.32). The extent of removal of residues from chilli fruits while dipping in water can be attributed to the difference in solubility of these insecticides in water and the different types of insecticide formulations. The acetamiprid and imidacloprid used in this experiment were water miscible formulations viz, SP (soluble powder) and SC (suspension concentrate) and water solubility of these insecticides at 21°C are comparatively high viz, 0.43g/l and 0.51g/l. The organophosphate insecticides viz, ethion and dimethoate are emulsifiable concentrates (EC) and water miscibility of these formulations were low. The new generation insecticides, viz, acetamiprid, imidacloprid, and spiromesifen were easily dislodged by water washing compared to organophosphate insecticides viz, ethion and dimethoate.

After five days of spraying, the extent of removal of acetamiprid residues from chilli fruits was maximum (95.89%), followed by spiromesifen (58.86%), ethion (27.38%), and propargite (27%) (Table.33). Among the different insecticides used in this experiment, spiromesifen, propargite and ethion are having contact action while imidacloprid, acetamiprid and dimethoate are having systemic action. At five days after spraying, because of the high systemic nature of imidacloprid and dimethoate, the penetrated residues on chilli fruits were not removed. The inference we can arrive from

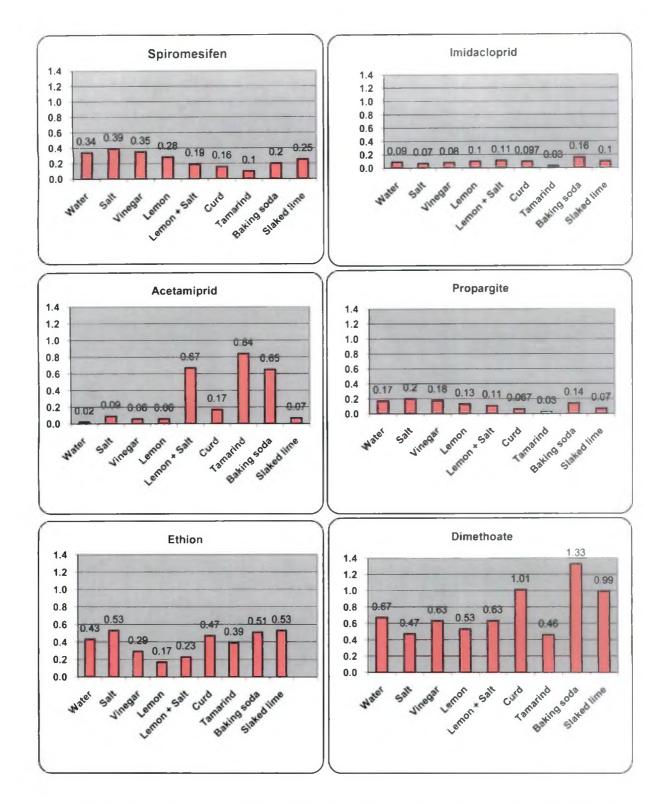


Fig. 18. Extent of removal of insecticide residues from chilli fruits expressed as processing factor using different treatment solutions after 2 hours of spraying. X- axis - Treatments, Y- axis - Processing factor

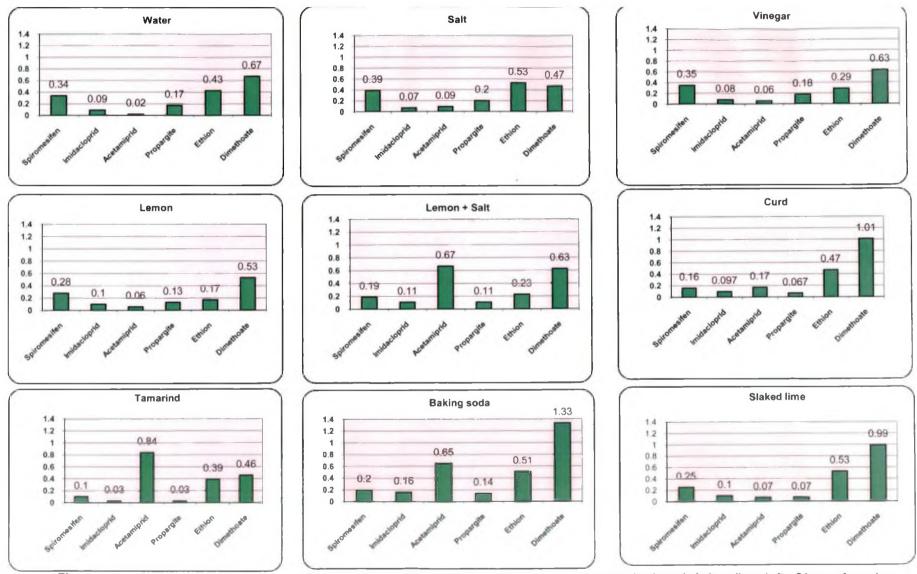
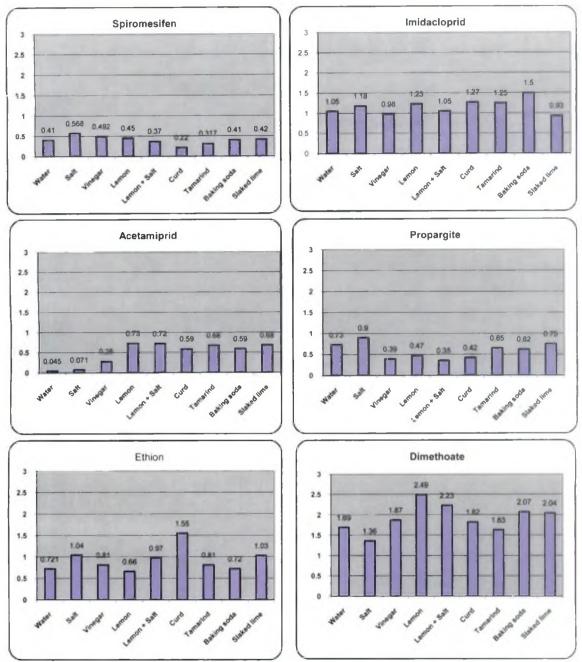
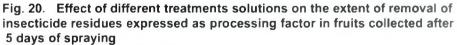


Fig. 19. Effect of different treatment solutions on the extent of removal of insecticide residues expressed as processing factor in fruits collected after 2 hours of spraying. X-axis- Insecticides, Y axis- Processing factor. the negative percentage values in the present study is that the surface residues may not be there over the fruits after five days of spraying and the penetrated residues cannot be removed by different washing techniques.

There are many reports where washing reduces the pesechniques.ticide residues from fruits and vegetables, but the literature pertaining to the decontamination of new class of insecticides from fruits and vegetables are scanty. Washing is the preliminary cleaning step in both household and commercial preparations. Loosely held residues of several pesticides are removed with a reasonable efficiency by varied types of washing processes (Street, 1969). The removal of pesticide residues by washing has also been found to depend on the age of the chemical, newly sprayed chemicals are easier to remove than those that have stayed on the crop longer. This may be the possible reason of the lesser extent of removal of insecticide residue on the 5<sup>th</sup> day of spraying compared to the day of spraying. Washing of soybeans twice with water reduced the pesticides by 80-90% of the initial levels of 5.01 ppm dichlorvos, 7.9 ppm malathion, 11.2 ppm chlorpyriphos and 2.87 ppm captan (Miyahara and Saito, 1994). Washing of okra fruits with tap water removed cypermethrin residues to the extent of 41.2-48.3% in 0 day samples and 37.1 to 46.0 in 5<sup>th</sup> day samples. In the case of fluvalinate, the extent of removal was 38.0-44.2% on 0 day and 32.4-41.8% on fifth day samples. (Singh et al, 2004b). Maximum (77%) reduction of organophosphate insecticides was observed in brinjal, followed by 74% in cauliflower and 50% in okra by washing (Kumari, 2008).

When the insecticide treated chilli fruits were subjected to dipping in salt solution 2% for twenty minutes followed by washing in water, maximum residues were removed in the case of imidacloprid (93.37%) sprayed chilli fruits followed by acetamiprid (91.15%), propargite (79.64%), spiromesifen (61.93%), dimethoate (52.67%) and ethion (48.17%) (Table. 32). The salt solution was found as a better option in the case of dimethoate sprayed chilli





X- axis - Treatments, Y- axis - Processing factor

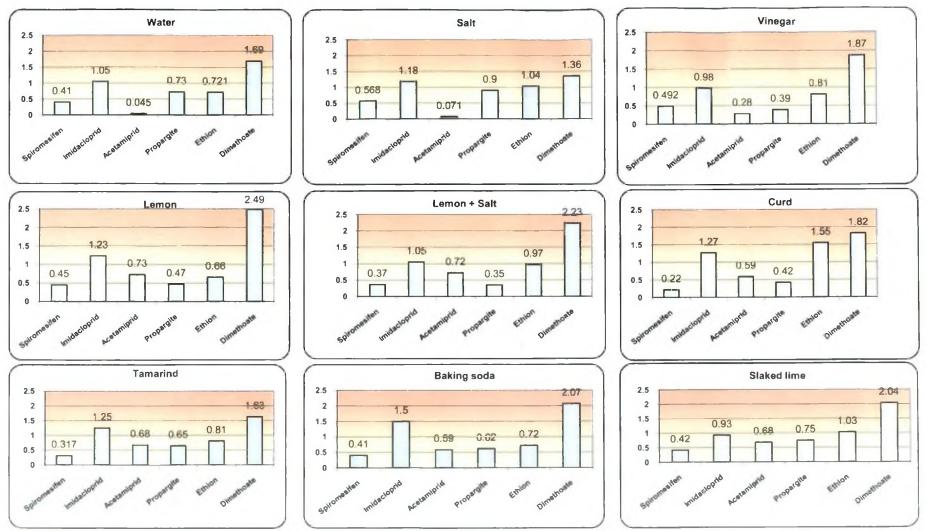


Fig. 21. Effect of different treatments solutions on the extent of removal of insecticide residues expressed as processing factor in fruits collected after 5 days of spraying . X axis- Insecticides Y axis- Processing Factor

fruits as it removed about twenty per cent more residues when compared to simple water wash at two hours after spraying. When the chilli fruits were dipped in salt solution 2% the ease of removing insecticide residues was more in the case of new insecticide class viz, neonicotinoids (acetamiprid, imidacloprid), keto-enase group (spiromesifen) and propargite when compared to conventional organo phosphate insecticides (ethion and dimethoate). At 5DAS, the extent of removal was maximum in the case acetamiprid (92.5%) followed by spiromesifen (43.10%) and propargite (20%), while in the case of imidacloprid, ethion and dimethoate sprayed chilli fruits, dipping in salt solution followed by washing in water was found to be ineffective (Table. 33)

Kumar (1997) reported that dipping of cow pea pods in 2% salt water for one hour removed more than 90% residues of monocrotophos and phosphamidon. In another study, dipping of grape berries in 2% salt solution for 10 minutes followed by washing with water proved to be an effective decontamination procedure, facilitating removal of 67.52 and 6.5 per cent of residues of chlorpyrifos and 51.77 and 50 per cent quinalphos residues, from fruits harvested at correspondingly at 1 and 5 day after spraying respectively (Reddy and Rao, 2002).

Dipping chilli fruits in vinegar 2% solution for twenty minutes followed by washing in water removed a greater extent of acetamiprid residues (94.23%) followed by imidacloprid (92.33%), propargite (88.29%), ethion (71.63%), spiromesifen (65.41%) and dimethoate (36.77%) in the decreasing order (Table 32). The percentage removal of the neonicotenoid group of insecticide was maximum in the vinegar treatment also and this is attributed by the polar nature of the insecticides belonging to this group. At 5 DAS, maximum removal was noticed in the case of acetamiprid (72.32%) followed by propargite (61%), spiromesifen (50.76%) and ethion (18.47%) (Table. 33). Two per cent vinegar solution is having pH in the range of 2-3, and it is clear from the results that the acidity of the vinegar solution is having

not much significance in the removal of insecticide residues as all the standed insecticides gave a fair removal of insecticide residues while dipping in water, but the extend of removal of the residues in the new generation insecticides like imidacloprid, acetamiprid, propargite and spiromesifen were more than OP compounds like dimethoate and ethion.

Dipping chilli fruits in 2% solution of lemon juice and a combination of lemon + salt 2% solution for twenty minutes followed by washing in water reduced imidacloprid residues by 90.05 % and 88.83 % respectively. The above two treatments reduced the residues of other insecticides in the descending order, propargite (87.27 % and 88.80 %), ethion (83.13 % and 77.51%), spiromesifen (72.51% and 80.96%), dimethoate (47.20% and 36.63%) and acetamiprid (42.86% and 33.46%) (Table 32). The lemon juice (2 %) solution removed more than 70 per cent of the residues of four insecticides viz, imidacloprid, propargite, ethion and spiromesifen tested in the present investigation. It is clear from the result that adding salt to lemon juice reduced the effectiveness of residue removal from chilli fruits. The results obtained in the present investigation revealed that lemon solution 2% solution can be considered as effective option for removing both conventional and new generation insecticides.

The two treatments viz, lemon 2% solution and lemon+salt 2% solution was not effective in removing the surface residues of imidacloprid and dimethoate at five days after spraying. The efficacy of the above two treatments in reducing the residues of the remaining four insecticides in the present study were in the order spiromesifen (54.92% and 62.80%), propargite (53% and 65%), ethion (33.76% and 2.55%) and acetamiprid (26.9% and 27.68%) (Table 33). The effectiveness of lime juice (2%) in reducing the residues of monocrotophos and phosphamidon in cow pea and bitter gourd was reported by Kumar (1997) where he recorded that dipping in 2% lemon juice for one hour removed more than 90% residues of monocrotophos and

phosphamidon in cow pea and bitter gourd, immediately after spraying with the insecticides.

About 93.38 per cent of propargite residues were removed when chilli fruits were dipped in 2 % curd solution for twenty minutes followed by washing in water. The extent of removal of insecticides in the case of all the new class of insecticides viz, spiromesifen, imidacloprid and acetamiprid were 84.36, 90.16 and 83.08 per cent respectively (Table 32). In the case of spiromesifen and propargite, there were 10- 20 per cent more removal than plain water wash. About 50% of the ethion residues were removed when fruits were dipped in curd solution followed by water washing. Dimethoate residues were not removed when the fruits were dipped in 2 % curd solution followed by water wash. At 5 DAS, 68.77, 58 and 45.54 per cent residues were removed in the case of spiromesifen, propargite and acetamiprid and the treatment had no effect on imidacloprid, ethion and dimethoate residues on chilli fruits (Table 33). Data obtained at zero day and 5 days after spraying, showed that dipping fruits in curd solution was a better option for removing spiromesifen residue from chilli fruits.

Dipping chilli fruits in tamarind solution 2% followed by washing in water removed fairly good amount of residues of imidacloprid (96.83%), propargite (96.69%), spiromesifen (90.03%), ethion (60.88%) and dimethoate (53.98%) (Table 32). As this treatment was found effective for majority of insects under the present study, tamarind solution can be recommended as a good decontaminating agent to remove pesticide residues from fruits and vegetables.Kumar (1997) reported the effectiveness of tamarind 2% solution in removing residues of phosphamidon and monocrotophos from bittergourd and cow pea pods. At 5 DAS, as in the previous cases the decontaminating agent had no effect on imidacloprid and dimethoate sprayed chilli fruits where as it removed 77.63, 30.5, 35 and 18.47 per cent residues from spiromesifen, acetamiprid, propargite and ethion sprayed chilli fruits (Table 33).

The two alkaline substrates used in the present investigations were baking soda and slaked lime. Among these two treatments, 2% solution of slaked lime was found to be slightly better than baking soda (2%) solution. Dipping chilli fruits in 2% slaked lime solution for twenty minutes followed by washing in water removed 93.28, 90.05, 75.07, 69.24, 46.70 and 0.65 per cent residues from propargite, imidacloprid, spiromesifen, acetamiprid, ethion and dimethoate sprayed chilli fruits respectively (Table. 32). In the baking soda treatment, the decreasing order of residue removal from chilli fruits were propargite (86.25%), imidacloprid (83.75%), spiromesifen (79.41%), ethion (48.90%) and acetamiprid (35.77%) (Table 32). Dimethoate sprayed chilli fruits were not decontaminated by dipping in slaked lime 2% solution for twenty minutes followed by washing in water. Both organophosphate insecticides in the present investigation viz, ethion and dimethoate get dislodged easily in acidic medium than in alkaline medium. But the new generation insecticides viz, spiromesifen, acetamiprid, imidacloprid and propargite did not show much difference in residue removal in acidic and alkaline medium. At 5DAS, dipping of chilli fruits in the alkaline treatments viz, baking soda 2% and slaked lime 2% solution removed 58.64% and 57.77% of spiromesifen residues respectively. When the chilli fruits were dipped in two per cent solutions of baking soda and slaked lime, 40.18% and 33.1% of acetamiprid residues and 38% and 25% of propargite residues were removed respectively (Table. 33). These alkaline treatments had negligible effect on the removal of imidacloprid, dimethoate and ethion residues at five days after spraying. There are reports that alkaline solution of 0.05% NaHCO3 was very effective in reducing residues of quinalphos and endosulfan in cauliflower (Senapathi et al., 1999). Washing in 10% sodium carbonate resulted in 92, 88, and 95 per cent removal of residues, while neutral solution of 10% sodium chloride removed only 42, 76 and 86 per cent of the residues of the insecticides respectively (Zohair, 2001).

From the above results it is not possible to recommend a single treatment for removing the pesticide residues from chilli fruits. The efficacies of the treatments differed with respect to different insecticidal chemistries. The effect of processing depends on many factors like, age of the chemical, water-octanol partition coefficients, water solubility, vapour pressure and heat stability. The effectiveness of washing also depend on the location of residue whereby surface residues are amenable to washing while the systematic residues present in the tissues will be affected only to a small extent (Holland et al., 1994). With an increasing time (age) the residues tend to move into cuticular waxes or deeper layers so the amount of residue that can be removed by washing declines. Water solubility of the insecticide reflects not only their higher solubility in wash but also their propensity to move in to wax layers (Kaushik, et al., 2009). On the fifth day of spraying the extent of removal of insecticides residues from chilli fruits was lower compared to the day of spraying and in the present investigation it is found that the residues of imidacloprid and dimethoate were not removed on the fifth day of spraying. Imidacloprid is having high water solubility (0.5 g/l at 20° C) thus providing the molecule a considerable mobility in the xylem of plants (Elbert et al., 1991). The two insecticides might have penetrated in to chilli fruits at five days of spraying. So it is better to avoid the use of systemic insecticides at the fruiting stage of the crop or if a compulsory situation arises the application of insecticides can be done after the plucking of mature fruits.

Pithily, the bio efficacy and safety of the different biorational insecticides used for the management of the sucking pest complex of chilli, *viz* mite, thrips and aphids were determined based on the investigations done at laboratory and field level. The efficacy of different insecticides against the sucking pests *vi.z*, mite and thrips were primarily evaluated at the laboratory in a confined environment adopting leaf disc methodology. The same chemicals were also evaluated against aphids released in to the chilli plants raised in Q tubs. Parallely these insecticide molecules were also evaluated against thrips

and mites in potted chilli plants. The safety of these insecticide molecules against the natural enemies of the sucking pest was also determined at the laboratory level. Detailing in to the result of laboratory evaluation, acetamiprid, spiromesifen, propargite, spinosad and dimethoate were found to be very effective against chilli mite whereas 100 per cent mortality of chilli thrips was recorded in spinosad, spiromesifen, imidacloprid, thiamethoxam, acetamiprid, propargite and dimethoate treated chilli leaf disc. The neonicotinoid insecticides viz, acetamiprid, imidacloprid, thiamethoxam and other insecticides viz, spiromesifen and dimethoate were found effective against chilli aphids. When the safety of these insecticides was studied, spiromesifen ranked first in their safety to coccinellid and hemerobid grubs. Flubendiamide, spirotetramat and indoxacarb were also found moderately safe to coccinellid grubs and hemerobid grubs. Dimethoate was found toxic to coccinellid and hemerobid grubs. Among the neonicotinoid insecticides, acetamiprid and thiamethoxam were found safer than imidacloprid. In the safety evaluation against Amblyseius spp, acetamiprid, thiamethoxam, spiromesifen and spirotetramat were found safer as it caused less than 70 per cent mortality after 72 hours. The results of the pot culture back up the laboratory evaluation where spiromesifen, imidacloprid and propargite were found to be effective against chilli mite and acetamiprid was found to be effective against chilli thrips. Lowest leaf curl index was recorded in spiromesifen and propargite sprayed chilli plants. The overall results of the above laboratory evaluation and pot culture study helped to identify the insecticide molecules like spiromesifen, imidacloprid, acetamiprid thiamethoxam, propargite and dimethoate as promising in their bio efficacy to sucking pests and their safety to the natural enemies and these selected molecules were further evaluated in field experiment.

In the field evaluation, two other insecticides registered nationally against sucking pest in chilli viz, ethion and oxy demeton methyl were also included as chemical checks. Among the different chemicals evaluated in field, the mite

population was significantly lower in propargite, spiromesifen and acetamiprid treated plots. Significantly lower population of chilli thrips was recorded in acetamiprid treated plots and lowest average leaf curl index (LCI) was recorded in spiromesifen and propargite treated plants. Thus it is proved that chilli leaf curling symptom was more due to chilli mites than chilli thrips. Regarding the safety of these insecticides to natural enemies in chilli ecosystem, viz coccinellids, predatory mite, spiders and other non targets, spiromesifen and propargite treated plots harboured more natural enemies than other insecticides, where as among the neonicotinoids, acetamiprid and thiamethoxam were comparatively safer than imidacloprid. Thus based on the field evaluation, all the insecticides evaluated for field evaluation except thiamethoxam were selected for the study of dissipation of the insecticides on chilli fruits. Thiamethoxam residues could not be measured for want of test facilities. As chilli is one of the major spice crop in India earning good foreign exchange, pesticide free commodity is an important aspect and can be accomplished only by observing sufficient pre harvest interval (PHI) / waiting period of the insecticides used in chilli pest management practices. Considering the waiting period of different insecticides in the present investigation, acetamiprid is having the shortest waiting period of 3 days whereas the conventional insectides like ethion and dimethoate recorded the maximum waiting period of 28 and 14 days. As chilli is a crop having short harvest interval, the application of insecticides should be as careful as possible and should be need based. Since the mite problem persist in chilli crop till the end of its fruiting period, application of acaricides and insecticides were inevitable, and if any detectable level of insecticide residues is present in the product, it will lead to the decline in the value of our produce in the international market.

In the case of spiromesifen maximum residue removal (90.03%) occurred when the fruits were dipped in tamarind 2% solution followed by washing. For imidacloprid, all the treatments were very effective in removing residues, more than eighty per cent of the residues were removed in all the treatments which justify its high polarity and the maximum removal was

recorded in tamarind treatment (96.83%). In the case of acetamiprid, mere water wash removed 97.69% of the residues where as treatments like lemon (42.86%), lemon + salt (33.46%), tamarind (16.08%) and baking soda (35.77%) were found less effective. In the case of propargite, maximum removal of residues was obtained by dipping in solution of tamarind (96.69%) and we can conclude that all the treatments were effective in removing residues. In the case of OP insecticides like ethion and dimethoate, the extent of removal of residues was less compared to the new generation insecticides like, spiromesifen imidacloprid, acetamiprid and propargite. Maximum extent of residue removal was observed when chilli fruits sprayed with ethion were dipped in lemon 2% solution (83.13%) followed by washing. The removal of dimethoate residues was lower than ethion and maximum residues were removed in tamarind (53.98%) treatment.

Out of six insecticides studied, dipping insecticide treated chilli fruits in 2% tamarind solution for twenty minutes followed by washing in water removed maximum amount of residues in the case of spiromesifen, imidacloprid, propargite and dimethoate. In the case of ethion also, tamarind treatment removed fairly good amount of residues. Owing to this majority, tamarind can be recommended as a good option for removing insecticide residues from fruits and vegetables.

## SUMMARY

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#### 6. SUMMARY

Chilli (*Capsicum annuum* L.) is one of the versatile spice and vegetable crops grown in India. As sucking pests is one of the major constraints in chilli production, laboratory and field trials were done to evaluate biorational insecticides for the management of sucking pests and to assess the safety of insecticides to their natural enemies. Chilli is one of the major spice crop in India earning good foreign exchange, pesticide free commodity is an important aspect which can be accomplished only by observing sufficient pre harvest interval (PHI) / waiting period of the insecticide used in pest management practices. With this in view, the dissipation patterns of seven different insecticides on chilli fruits were studied. Finally, studies were carried out to find the effect of different domestic decontamination practices which can reduce the pesticide load in chilli fruits. The results of the studies are summarized here under.

The efficacy of eight new generation insecticides viz., spinosad 75 g a.i. ha<sup>-1</sup>, spiromesifen 100 g a.i. ha<sup>-1</sup>, spirotetramat 60 g a.i. ha<sup>-1</sup>, indoxacarb 60 g a.i. ha<sup>-1</sup>, imidacloprid 20 g a.i. ha<sup>-1</sup>, thiamethoxam 40 g a.i. ha<sup>-1</sup>, flubendiamide 60 g a.i. ha<sup>-1</sup> and acetamiprid 20 g a.i. ha<sup>-1</sup> was assessed in comparison with propargite 570 g a.i ha<sup>-1</sup> and dimethoate 300 g a.i ha<sup>-1</sup> as acaricidal and insecticide check, against three sucking pests of chilli viz, mites, thrips and aphids under laboratory conditions.

- In the laboratory evaluation, the insecticides spiromesifen and acetamiprid caused 100 percent mortality of chilli mites at 24 hours after release of mites into the insecticide treated leaf disc. Observations were taken up to 72 hours of release and by the time cent per cent mortality was also recorded in spinosad, propargite and dimethoate treated leaf disc.
- The data on the effect of new generation insecticides on chilli thrips revealed that acetamiprid was highly toxic to chilli thrips and it is the only

chemical which caused 100 percent mortality of thrips after 24 hours of release in to the insecticide treated leaf disc. After 72 hours of release of thrips into insecticide treated leaf disc, 100 percent mortality was recorded in leaf discs treated with spinosad, spiromesifen, imidacloprid, thiamethoxam, acetamiprid, propargite and dimethoate.

• Laboratory screening of new generation insecticides against chilli aphids showed that neonicotenoid insecticides viz, acetamiprid and thiamethoxam recorded 100 percent mortality of chilli aphids after one day of spraying. At 3 DAS, another neonicotenoid insecticide viz, imidacloprid along with spiromesifen and dimethoate had recorded cent percent mortality of chilli aphids.

• The insecticides which were found not effective against mites, thrips and aphids based on the laboratory evaluation were spirotetramat, indoxacarb and flubendiamide.

• In the laboratory evaluation, spiromesifen was found very safe to coccinellid grubs and hemerobid larvae. It caused only 20 % mortality of coccinellid grubs and 40% mortality of hemerobid larvae even 72 hours after release of insect in to the insecticide treated leaf. Flubendiamide, spirotetramat and indoxacarb were also found moderately safe to coccinellid grubs and hemerobid larvae, but they were not effective against the pest complex. Dimethoate was found to be very toxic to coccinellid and hemerobid grubs. Among the neonicotenoid insecticides, acetamiprid and thiamethoxam were found safer to coccinellid and hemerobid grubs than imidacloprid.

• In the safety evaluation against *Amblyseius* spp, acetamiprid, thiamethoxam, spiromesifen and spirotetramat were found safer than other insecticides. 100 percent mortality of predatory mite was recorded in the acaricidal and insecticidal check viz, propargite and dimethoate respectively.

• In pot culture experiment, significantly lower population of chilli mites was recorded in spiromesifen, propargite, imidacloprid, acetamiprid, thiamethoxam and dimethoate sprayed chilli plants. Chilli thrips were not recorded in acetamiprid sprayed chilli plants upto 7 DAS. When the damage caused due to the feeding injury of mites and thrips were indexed, (Leaf curl Index) lower index was recorded in spiromesifen and propargite sprayed chilli plants.

• Based on the above results of the laboratory and pot culture studies, the insecticides selected for the field evaluation based on their bioefficacy against *P. latus*, *S. dorsalis*, and *A. gossypii* were spiromesifen, imidacloprid, thiamethoxam, acetamiprid, propargite and dimethoate. In addition to this, two organophosphate which are widely recommended and used for chilli cultivation were also included *viz.*, ethion and oxy demeton methyl.

• The insecticidal treatments were found effective in controlling mites and thrips population when compared to the untreated control. In the initial stage (vegetative) of the crop (30 DAT), the mite population was lower when compared with population of thrips population. The mite population was significantly lower in propargite, spiromesifen and acetamiprid treated plots. The next best treatments in their decreasing order of efficacy against chilli mite were imidacloprid, thiamethoxam, oxy demeton methyl, dimethoate and ethion.

• The treatments in the decreasing order of efficacy in controlling chilli thrips were acetamiprid, thiamethoxam, spiromesifen, oxy demeton methyl, imidacloprid, dimethoate, ethion and propargite. The order of efficacies of the insecticides in controlling chilli mites and thrips remained same in the three consecutive insecticidal sprays given at 30DAT, 60 DAT and 90 DAT. • Lowest average leaf curl index (LCI) was recorded in spiromesifen, propargite, acetamiprid and imidacloprid and statistically superior to other treatments. All the insecticidal treatments were significantly superior over the untreated control. In general the treatments given at 30, 60 and 90 days after transplanting suppressed the population of the two major sucking pests viz, *S. dorsalis* and *P. latus* without allowing to reach economic injury level.

• In the field evaluation, significantly more number of predatory mites was recorded from spiromesifen treated plots when compared to other insecticide treated plots whereas propargite, dimethoate and imidacloprid treated plots were devoid of any predatory mites after application of insecticides, indicating them as not suitable to be integrated with pest management in chilli.

• Then coccinellid beetles were not much affected by the application of spiromesifen and propargite, acetamiprid and thiamethoxam were found moderately safer to coccinellid beetles.

• Spider populations were not found in many of the insecticide treated plots up to 5th day after spraying. But population existed in spiromesifen treated plots and on the 14<sup>th</sup> day of spraying the spider population in spiromesifen treated plots came equal with those in untreated plots.

• The neonicotenoid insecticides, acetamiprid and thiamethoaxam were also observed as selective insecticides when compared to imidacloprid and other conventional insecticides like ethion, dimethoate and oxy demeton methyl which harboured less number of natural enemies.

• Spiromesifen treated chilli plants recorded maximum plant height when compared to other insecticidal treatments. Maximum number of terminal branches was recorded in propargite treated chilli plants followed by spiromesifen and all the insecticide treated plants were statistically superior to the untreated plants. The yield of chilli was more in spiromesifen treated plants followed by acetamiprid sprayed chilli plants.

• The analytical procedure gave good recovery of spiromesifen residues, 85 - 94 % from chilli fruits when fortified at concentration ranging from  $0.01 - 0.5 \text{ mg kg}^{-1}$ . A calibration curve was prepared by plotting different concentrations (0.01, 0.05, 0.1, 0.5 and 1 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.01-1mgkg<sup>-1</sup> concentration. Spraying of spiromesifen @ 100g a.i ha<sup>-1</sup> on chilli fruits deposited an initial average deposit 0.609 mg kg<sup>-1</sup>. On the seventh day about 95.1 % of the initial residues were degraded and on the tenth day the residues were below detectable level. The residues of spiromesifen on chilli fruits declined progressively with time and the half life calculated from the regression equation was found to be 1.71 days. The waiting period of spiromesifen on chilli fruits was calculated as 7.03 days.

• The analytical procedure gave good recovery of imidacloprid residues, 88 - 113 % from chilli fruits when fortified at concentration ranging from  $0.01 - 0.1 \text{ mg kg}^{-1}$  A calibration curve was prepared by plotting different concentrations (0.05, 0.1, 0.2, 0.3 and 0.4 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.05 - 0.4 mg kg<sup>-1</sup> concentration. Spraying of imidacloprid 17.8 g a.i. ha<sup>-1</sup>, left an initial average residue of 1.27 mg kg<sup>-1</sup> on chilli fruits. The residue of imidacloprid was below detectable level on the tenth day of spraying. The dissipation data followed first order kinetics and the half life calculated from the regression equation was 2.08 days. The residue of imidacloprid was below detectable level on the tenth day of spraying. Based on the MRL value of 0.03 mg kg<sup>-1</sup> fixed by PFA in India, a waiting period of 6.8 days was calculated for imidacloprid on chilli.

• The recovery of acetamiprid for the selected analytical procedure on chilli fruits was in the range of 90.06 to 100.16 per cent at the concentration

of 0.01 to 2.5mg kg<sup>-1</sup>. Good linearity was found within the range of 0.4 - 1 mg kg<sup>-1</sup> concentration. The initial residues of acetamiprid on chilli fruits at the time of spraying was at the level of 2.44 mg kg<sup>-1</sup>. The residue of acetamiprid was below detectable level on  $20^{th}$  day of spraying. In the present study the half life (T <sub>1/2</sub>) of acetamiprid on chilli fruits calculated from the regression equation were found to be 2.27 days. Based on the MRL of 0.3 mg kg<sup>-1</sup> fixed by European Union, a waiting period of 3.51 days is recommended for the safe consumption of acetamiprid sprayed chilli fruits.

• The analytical procedure gave good recovery of propargite residues, 91.33, 85.67 and 91.67 per cent at 0.01, 0.05 and 0.1mg kg<sup>-1</sup> respectively. A calibration curve was prepared by plotting concentrations (0.1, 0.5, 1.0 and 2.0 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.1-2.0 mgkg<sup>-1</sup> concentration. On the day of spraying, the chilli fruits collected at 2hrs after spray recorded an average initial deposit of 1.54 mg kg<sup>-1</sup> propargite residues. From the dissipation data a half -life of less than one day was calculated for propargite and a waiting period of six days was recommended for propargite based on the MRL value (2 mg kg<sup>-1</sup>) prescribed by the European Union.

• When chilli fruits were fortified with 0.01, 0.1, 0.5 and 1.0 mg kg<sup>-1</sup> of dimethoate, the mean recovery percentage were 73.4, 95.7, 80.3 and 89.16 respectively. A calibration curve was prepared by plotting concentration (0.01, 0.1 and 1 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.01-1 mg kg<sup>-1</sup> concentration. On the day of spraying, an initial deposit of 7.36 mg kg<sup>-1</sup> of dimethoate was recorded on chilli fruits. From the dissipation data, the half life (T<sub>1/2</sub>) values of dimethoate were 1.94 days. MRL has been fixed as 0.5 mg kg<sup>-1</sup> for dimethoate in India. Based on the MRL values a waiting period of 13.63 days was calculated for dimethoate.

• The recovery percentage of ethion for various fortification levels at 0.01, 0.1, 0.25 and 0.5 mg kg<sup>-1</sup> were 94.9, 76.4, 86.21 and 77.25 respectively. A calibration curve was prepared by plotting different concentration (0.01, 0.1, 0.5 and 1.0 mg kg<sup>-1</sup>) vs. peak area. Good linearity was found within the range of 0.01 - 1.0 mg kg<sup>-1</sup> concentration. The residues were below detectable level on the twentieth day of spraying. Based on the dissipation data, the half life (T<sub>1/2</sub>) values of ethion was 3.43 days .The MRL value has been fixed as 0.1 mg kg<sup>-1</sup> for ethion and based on this value, a waiting period of 28 days was calculated for ethion.

• The recovery percentage of oxy demeton methyl at concentrations, 0.03, 0.1 and 0.5 mg kg<sup>-1</sup> were 76.01, 89.66 and 94.66 percentage respectively. On the day of spraying, the chilli fruits recorded an initial deposit of 0.966 mg kg<sup>-1</sup>. The residues were below detectable level on fifteenth day of spraying. Based on the dissipation data a half life of 4 days was calculated for oxy demeton methyl. Based on the MRL value (0.01 mg kg<sup>-1</sup>) fixed by the European Union (EU), a waiting period of 7 days has been calculated in chilli.

• Of the various insecticides studied in the present investigation, acetamiprid is having the shortest waiting period (3.51 days) and ethion was having the longest waiting period (27.89 days). In chilli usually plucking is done at an interval of 5- 6 days interval and if the farmers are not strictly observing appropriate waiting periods for plucking chilli fruits, the residues of spiromesifen, imidacloprid, propargite dimethoate, ethion and oxy demeton methyl could be a problem considering food safety aspects.

The insecticides selected to study the effect of different domestic processing techniques in removing the insecticide residues on chilli fruits harvested at two hours after spraying and at five days after spraying were spiromesifen, imidacloprid, acetamiprid, propargite ethion and dimethoate.

• In the case of spiromesifen maximum residue removal (90.03%) occurred when the fruits were dipped in tamarind 2% solution followed by washing with water where as only 62 per cent of the spiromesifen residues could be removed when the chilli fruits were dipped in 2% salt solution followed by washing in water. The same trend followed in the removal of residues from fruits harvested at five days after spraying (DAS) where dipping in tamarind solution followed by washing in water was the best treatment.

• For imidacloprid, all the treatments were very effective in removing residues, more than eighty per cent of the residues were removed in all the treatments could be attributed to the high polarity of the chemical and the maximum removal was recorded in tamarind treatment (96.83%) and least residues were removed when the chilli fruits were dipped in 2% solution of baking soda for followed by washing in water. Imidacloprid is a highly systemic insecticide and its residue concentration was very low at 5 DAS. Greater extent of penetration into the fruit wall might be the reason for lesser extent of decontamination.

• In the case of acetamiprid, mere water wash removed 97.69 per cent of the residues where as treatments like lemon (42.86%), lemon+salt (33.46%), tamarind (16.08%) and baking soda (35.77%) were found less effective. At 5 DAS also, the extent of removal was maximum when the fruits were dipped in water for twenty minutes followed by washing in water.

• In the case of propargite, maximum removal of residues was obtained by dipping the chilli fruits in 2% solution of tamarind (96.69%) for twenty minutes followed by washing in water and dipping fruits in 2 % salt solution followed by washing in water removed 79.64 per cent of the residues which recorded the lowest removal among the different

treatments. At 5 DAS, the extent of removal was maximum when the fruits were dipped in 2% solution of combination of lemon+salt solution.

• The extent of residue removal of OP insecticides viz, ethion and dimethoate was less when compared with the case of other new generation insecticides in the present investigation. In the case of ethion sprayed chilli fruits, maximum residues were removed when the fruits were dipped in 2% solution of lemon juice and only 46.7 per cent of the residues got removed when the fruits were dipped in 2% solution of slaked lime which was found as the least effective treatment to remove residues of ethion. The trend in the residue removal of ethion remained same at 5 DAS. For dimethoate sprayed chilli fruits, maximum removal of residues occurred when they were dipped in 2% solution of tamarind and treating chilli fruits in 2% solution of curd had no effect in removing dimethoate residues from chilli fruits. As dimethoate is a highly systemic insecticide, the removal of insecticide residues after five days was not at all effective.

• Dipping insecticide treated chilli fruits in 2% tamarind solution for twenty minutes followed by washing in water removed maximum amount of residues in the case of spiromesifen, imidacloprid, propargite and dimethoate. In the case of ethion also, tamarind treatment removed fairly good amount of residues. Owing to this majority, dipping in tamarind solution (2%), can be recommended as a good option for removing insecticide residues from fruits and vegetables.

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### BIOEFFICACY AND SAFETY EVALUATION OF BIORATIONAL INSECTICIDES FOR THE MANAGEMENT OF SUCKING PEST COMPLEX OF CHILLI (CAPSICUM ANNUUM L.)

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#### Abstract of thesis submitted in partial fulfillment of the requirement

for the degree of

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#### ABSTRACT

The efficacy and safety of biorational insecticides used for the management of sucking pest complex of chilli, viz. mites (*Polyphagotarsonemus latus* Banks), thrips (*Scirtothrips dorsalis* Hood) and aphids (*Aphis gossypii* Glover) were tested in laboratory and field conditions.

The efficacy of eight new generation insecticides *viz.*, spinosad 75 g a.i. ha<sup>-1</sup>, spiromesifen 100 g a.i. ha<sup>-1</sup>, spirotetramat 60 g a.i. ha<sup>-1</sup>, indoxacarb 60 g a.i. ha<sup>-1</sup>, imidacloprid 20 g a.i. ha<sup>-1</sup>, thiamethoxam 40 g a.i. ha<sup>-1</sup>, flubendiamide 60 g a.i. ha<sup>-1</sup> and acetamiprid 20 g a.i. ha<sup>-1</sup> was assessed in comparison with propargite 570 g a.i ha<sup>-1</sup> and dimethoate 300 g a.i ha<sup>-1</sup> as acaricidal and insecticide check, against three sucking pests of chilli viz, mites, thrips and aphids under laboratory conditions.

Based on the laboratory evaluation, acetamiprid, spiromesifen, propargite, spinosad and dimethoate were very effective against chilli mite whereas 100 percent mortality of chilli thrips was obtained in spinosad, spiromesifen, imidacloprid, thiamethoxam, acetamiprid, propargite and dimethoate in leaf disc method. The neonicotinoid insecticides viz, acetamiprid, imidacloprid, thiamethoxam and other insecticides viz, spiromesifen and dimethoate were found effective against chilli aphids. Spiromesifen was found as the safest insecticide to coccinellid and hemerobid grubs. Flubendiamide, spirotetramat and indoxacarb were also found safe to coccinellid grubs and hemerobid grubs, but they were not effective against the pest complex of chilli. Dimethoate was found toxic to coccinellid and hemerobid grubs. Among the neonicotinoid insecticides, acetamiprid and thiamethoxam were safer than imidacloprid. In the safety evaluation against the predatory mite *Amblyseius* spp, acetamiprid, thiamethoxam, spiromesifen and spirotetramat were safer than other insecticides.

In the pot culture study, significantly lower population of chilli mites were recorded in spiromesifen, propargite, imidacloprid, acetamiprid, thiamethoxam and dimethoate sprayed chilli plants. Chilli thrips were not recorded in acetamiprid sprayed chilli plants upto seven days after spraying. When the damage caused due to the feeding injury of mites and thrips were indexed, (Leaf curl Index) lower indices were recorded in spiromesifen and propargite sprayed chilli plants.

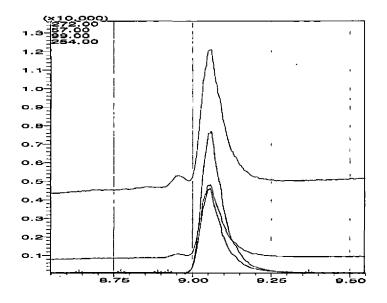
Among the different chemicals evaluated in field viz, spiromesifen, imidacloprid, acetamiprid thiamethoxam, propargite and dimethoate, ethion and oxy demeton methyl, the mite population was significantly lower in propargite, spiromesifen and acetamiprid treated plots. Acetamiprid treated plots recorded the lowest thrips population and lowest average leaf curl index (LCI) was recorded in spiromesifen and propargite treated plants. Spiromesifen and propargite treated plots harboured maximum numbers of natural enemies, where as among the neonicotinoids, acetamiprid and thiamethoxam were comparatively safer than imidacloprid. The yield of chilli was more in spiromesifen treated plants followed by acetamiprid sprayed chilli plants.

Considering the waiting period of different insecticides worked out in the present investigation, acetamiprid is having the shortest waiting period of 3.51 days and it is the only insecticide which fits well in to the harvest interval of chilli fruits, whereas the conventional insectides like ethion and dimethoate recorded the maximum waiting period of 27.89 and 13.63 days, respectively. The half-life of acetamiprid, ethion and dimethoate were 2.27, 3.43 and 1.94 days respectively. The insecticide spiromesifen, sprayed on chilli fruits had a waiting period of 7.03 days to reach below the MRL of 0.5 ppm and the time taken for half of the spiromesifen to degrade was 1.71 days. Imidacloprid sprayed on chilli fruits took 2.08 days to degrade its residues to half of the initial deposit and the waiting period was fixed as 6.8 days. Propargite had a waiting period of 5.7 days on chilli fruits and the half life was calculated as 0.63 days. The insecticides selected to study the effect of different processing techniques in removing the insecticide residues on chilli fruits harvested at two hours after spraying and at five days after spraying were spiromesifen, imidacloprid, acetamiprid, propargite, ethion and dimethoate.

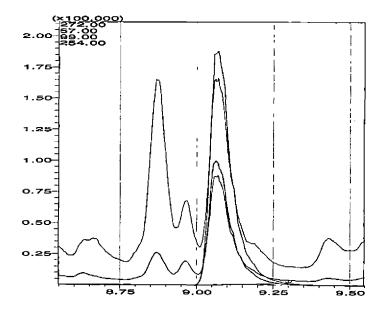
In the case of spiromesifen, maximum residue removal (90.03%) occurred when the fruits were dipped in tamarind 2% solution followed by washing in water. For imidacloprid, all the treatments were very effective in removing residues, more than eighty percent of the residues were removed in all the treatments which justify its high polarity and the maximum removal was recorded when the fruits were dipped in 2% solution of tamarind (96.83%) followed by washing in water. In the case of acetamiprid, mere water wash removed 97.69% of the residues where as in the case of propargite, maximum removal of residues was obtained by dipping in 2% solution of tamarind (96.69%) for twenty minutes followed by washing in water. The extent of residue removal in the OP insecticides viz, ethion and dimethoate was less when compared to other new generation insecticides. In the case of ethion sprayed chilli fruits, maximum residues were removed when the fruits were dipped in 2% solution of lemon juice (83.13%) followed by washing in water. For dimethoate sprayed chilli fruits, maximum removal of residues occurred when the fruits were dipped in 2% solution of tamarind (53.98%).

Out of six insecticides studied, dipping insecticide treated chilli fruits in 2% tamarind solution for twenty minutes followed by washing in water removed maximum amount of residues in the case of spiromesifen, imidacloprid, propargite and dimethoate. In the case of ethion also, tamarind treatment removed fairly good amount (60.88%) of residues. Owing to this majority, tamarind can be recommended as a good option for removing insecticide residues from fruits and vegetables.

Appendix - I

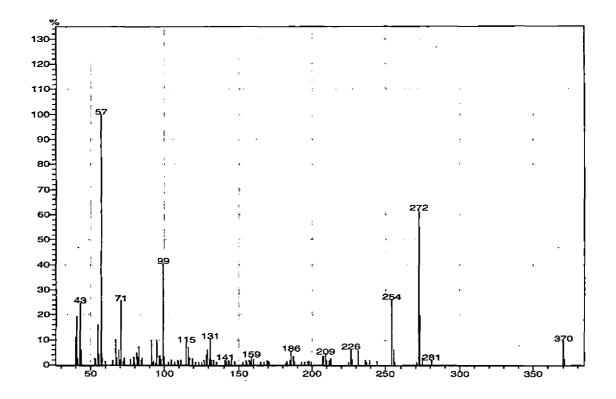


Chromatogram. 1a. Certified reference material of spiromesifen 1ppm

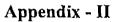


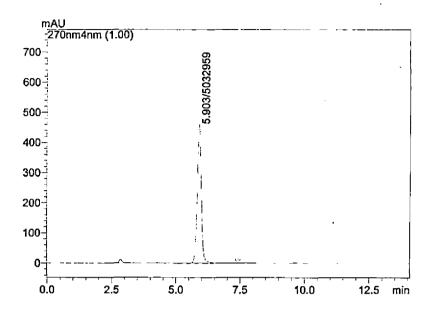
Chromatogram. 1b. Spiromesifen in chilli matrix

# Appendix - IA

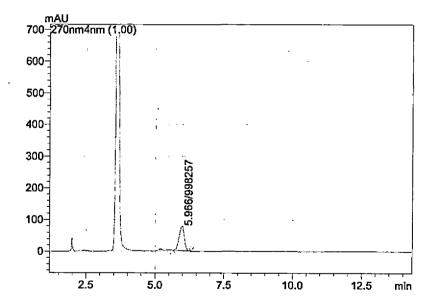


Mass spectrum of spiromesifen

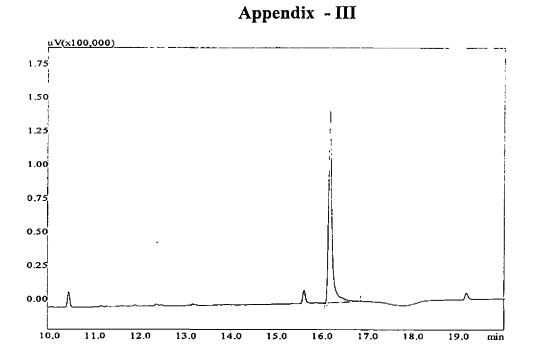




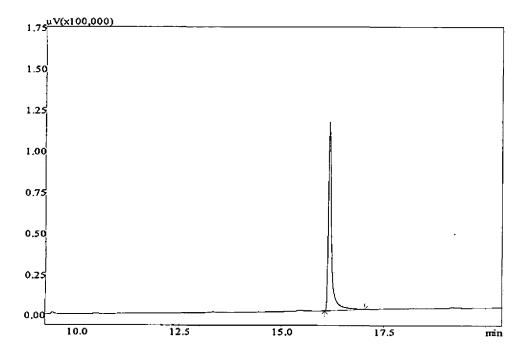
Chromatogram .2a. Certified reference material of imidacloprid 0.1ppm



Chromatogram 2b. Imidacloprid in chilli matrix

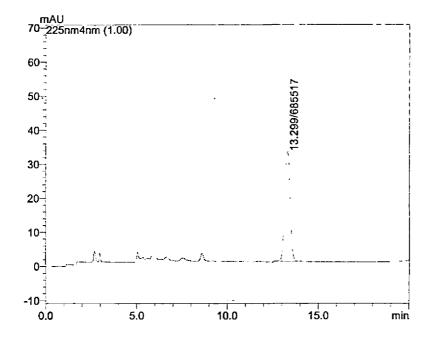


hromatogram. 3a. Certified reference material of acetamiprid 1ppm

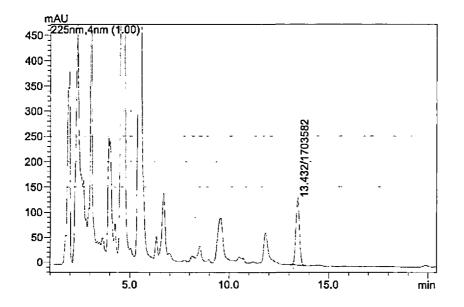


hromatogrtam.3b. Acetamiprid in chilli matrix

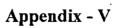
## Appendix - IV

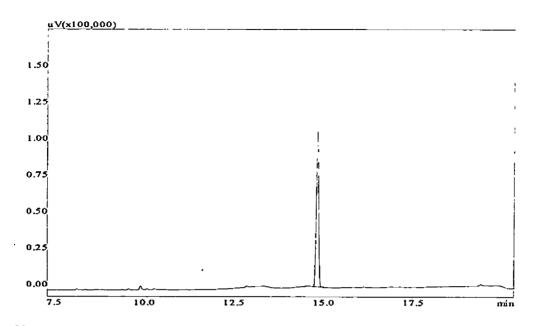


Chromatogram .4a. Certified reference material of propargite 5 ppm

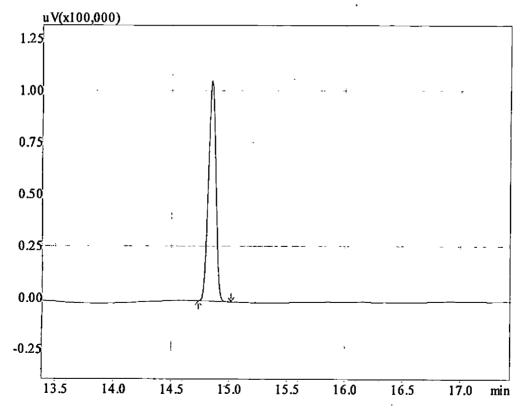


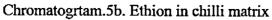
Chromatogram 4b. Propargite in chilli matrix

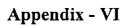


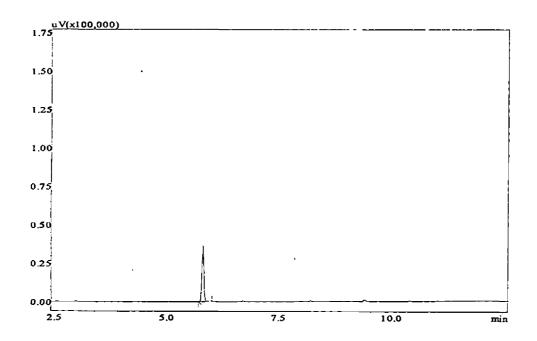


Chromatogram. 5a. Certified reference material of ethion 1ppm

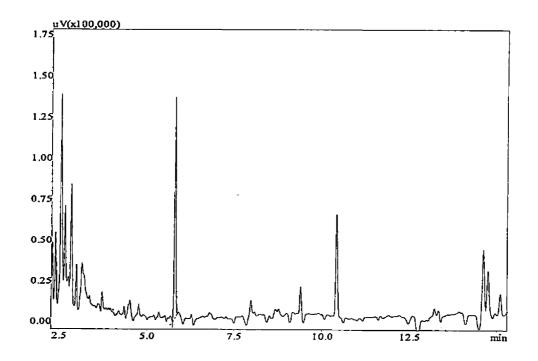






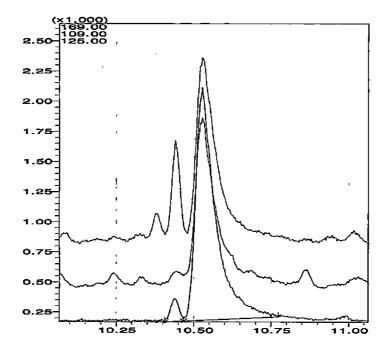


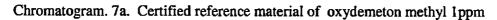
Chromatogram. 6a. Certified reference material of dimethoate 1ppm

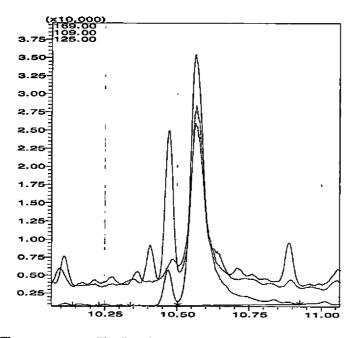


Chromatogrtam.6b. Dimethoate in chilli matrix

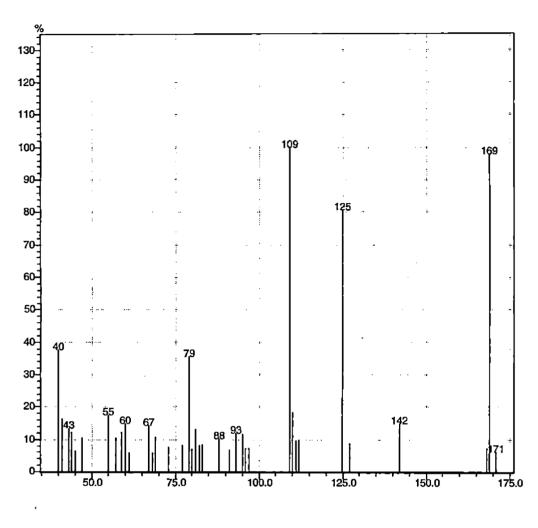








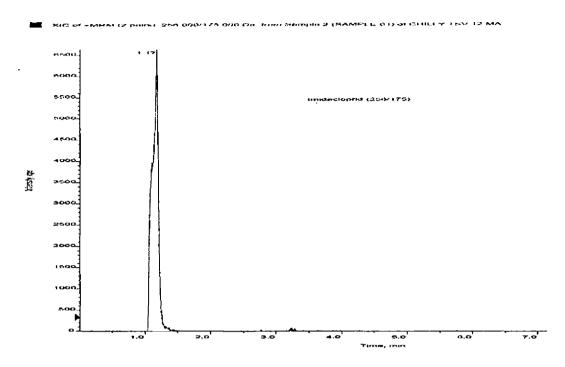
Chromatogram. 7b. Oxydemeton methyl in chilli matrix

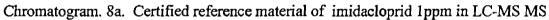


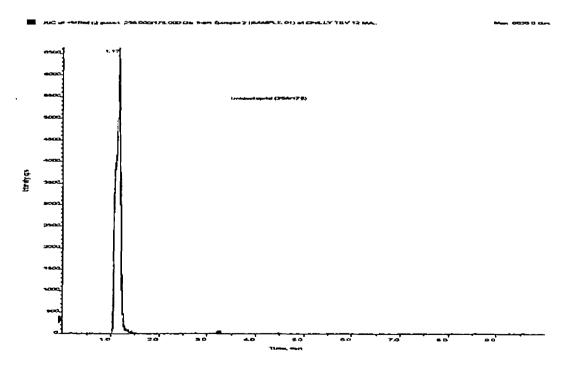
Appendix - VII A

Mass spectrum of Oxydemeton methyl

### Appendix - VIII

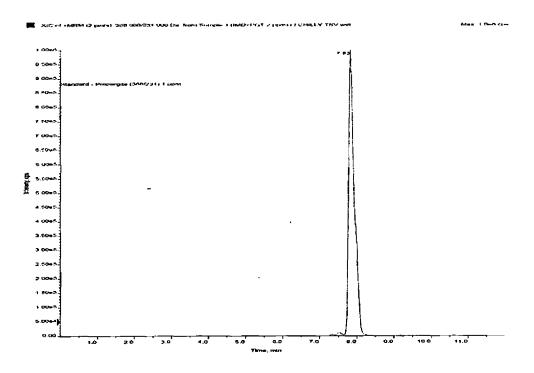


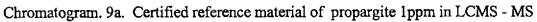


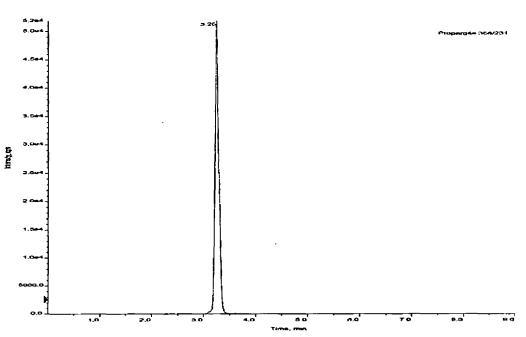


Chromatogram. 8b. Imidacloprid in chilli matrix (LCMS-MS)

## Appendix - IX







Chromatogram. 9b. Propargite in chilli matrix (LCMS-MS)

