234IMPACT OF PESTICIDES ON ABIOTIC AND BIOTIC COMPONENTS IN RICE ECOSYSTEM OF KUTTANADU

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DECLARATION

I hereby declare that this thesis entitled '**Impact of pesticides on abiotic and biotic components in rice ecosystem of Kuttanadu'** is a bonafide record of research work done by me during the course of research and that the thesis has not previously formed the basis for the award of any degree, diploma, associate ship, fellowship or other similar title, of any other university or society.

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Dedicated to my

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INTRODUCTION

1. INTRODUCTION

Rice is the staple food of more than 95 per cent of population in Kerala. Kuttanadu, the rice bowl of Kerala is unique among the rice ecologies of the world; the biggest wetlands of the country, located 0.5 – 2.5 metres below mean sea level (MSL). The history of the paddy cultivation in Kuttanadu can be traced back to centuries. The evolution of paddy cultivation in Kuttanadu was correlated to the technological advancement and changes in the regulatory frame work existed during the 19th and 20th centuries. In the earlier times, the reclamation was done mainly from the shallow part of the Vembanad lake or from the periphery of river Pamba. Rice is grown by construction of bunds and dewatering the so formed polders mainly during the puncha season from October - November to January - February. The soils of Kuttanadu are low to medium in fertility. Soil is enriched by annual silt deposition during the monsoon floods. The soils are alluvial with silty clay texture and are acid sulphate in nature with excessive iron content. The major problems faced by Kuttanadu rice are flood and lack of drainage, intrusion of saline water and soil acidity. Prevention of saline water by Thannermukkom Regulator affected the discipline in the season of rice cultivation, which along with the use of fertilizer responsive high yielding varieties with less resistance to pests and diseases, high seed rate, non-judicious fertilizer application and plant protection measures have polluted the system. In addition to the above the rice cultivation in Kuttanadu faces ever so many other socio-economic problems, just as in most of the other parts of the State. In spite of the sharp decline in area under rice; Kuttanadu rice bowl (53600 ha.) accounts for 18 per cent of the rice growing area and 25 per cent of total production of the State.

Rice ecosystem in Kuttanadu is unique in all features with no comparison elsewhere in the world. Kuttanadu lies 0.5-2.5 m below mean sea level and rice is the only crop that can be cultivated in most parts of the region. Water is drained off the field regularly using indigenous pump 'Petti and Para'to avoid submergence. The main crop growing season is 'Puncha' which corresponds to the summer season in Kerala. Due to unique climatic conditions in Kuttanadu, the rice crop is prone to infestation by several insect pests, diseases and weeds which necessitates regular and frequent use of large quantity of pesticides.

The indiscriminate and frequent use of pesticides may directly or indirectly affect the biotic and abiotic factors of the eco-system. A few studies have been so far conducted relating to the indirect effects of pesticides in Kuttanadu rice eco-system.

When pesticides are applied on rice plants, many droplets fall between the foliage especially in the inter row spaces and contaminate the irrigation water and soil (Mathew, 1982). When water is drained off from the fields to the canals the pesticide molecules along with water will reach adjacent canals, streams, rivers and finally the lakes. The pesticide molecules undergo several transformations leading to the formation of more water soluble forms. These molecules will be present in association with sediments, suspended particles or dissolved in water. In Kuttanadu frequent draining of water from rice field may lead to the discharge of pesticide molecules to near by channels and from there to stream and then to river or reservoir. This type of contamination is termed as non-point source of contamination. Many factors prevailing in the wet land eco-system influence the fate and transport of pesticide molecules and its contribution to non-point source of contamination.

The fate of pesticides applied in agricultural ecosystem is governed by the transfer and degradation processes and their interactions. Transfer is physical process in which the pesticide molecules remain intact, it includes sorption-desorption, runoff, percolation, volatilisation and absorption by crop plants or animals. Degradation, a chemical process in which pesticide molecules are split, includes photodecomposition, microbiological decomposition, chemical decomposition and plant detoxification transfer and degradation. Transfer and degradation determine pesticide persistence or retention, its efficacy for pest control as well as its potential for contamination of soil and water resources (Roger and Bhuhian, 1993).Several reports indicated that pesticides were degraded slowly in acid sulphate soils which is the predominant soil group in Kuttanadu. This was observed for gamma and beta BHC (Siddaramappa and Sethunathan, 1975) and endrin (Gowda and Sethunathan, 1976) and carbofuran (Venkateswararlu and Sethunathan, 1984).

Among the different processes, run-off, the lateral movement of water on land surface, is mainly responsible for transport of pesticide molecules from target area to adjacent water bodies. Run-off results from excessive application of irrigation water or rainfall or from draining of the field to get rid of the water. Pesticides in run-off water from rice fields may be both in suspension and as adsorbed in colloidal particles. The time interval between the application of the pesticides and the irrigation sufficient to produce runoff has significant effect on the amount of pesticide transported by the run off. Rainfall would wash off pesticide sticking to canopy and other parts of the plant and move it to the standing water on the surface from where it may be transported to run off. Runoff water from rice fields ultimately is discharged into large bodies of water such as lake, river or sea. At the IRRI experimental farm organophosphates and carbamates in run off water were found to be on the average at 1.0 ppb with a range of 0 to 20 ppb (IRRI, 1998). Monitoring of pesticide residues in river systems in Japan indicated the presence of a number of herbicides commonly used in paddy fields (Vu *et al*., 2006).

Very few studies had been conducted in Kuttanadu to estimate the quantity of pesticide loss through run off and the extent to which it contaminate the ecosystem. For suggesting feasible remedial measures, data on dynamics of pesticide residues in wet land ecosytem of Kuttanadu is necessary. Hence the present study was aimed with the following objectives

1. To study the pesticide use pattern and extent of contamination in rice ecosystem of Kuttanadu
2. To assess the relative impact of conventional pest control and IPM practices
3. To suggest measures for minimizing the residue hazards.

REVIEW OF LITERATURE

2. REVIEW OF LITERATURE

Pesticide application is currently the most widely practiced method of pest control in rice and rice based cropping system. The pesticides applied on rice crop may directly or indirectly contaminate the biotic and abiotic components of rice ecosystem. The literature on studies on pesticide consumption and contamination in rice, dynamics of pesticide residues in rice ecosystem and the effect of residues on various components in the ecosystem are reviewed herewith.

2.1 PESTICIDE USE PATTERN IN RICE

Rice crop suffers heavy losses on account of infestation by insect pests, diseases and weeds and a number of pesticides have been recommended to combat these problems (KAU, 2007).

Rice farmers often resorted to over application of pesticides and tried irrational combinations of pesticides to face the problem of attack by insect pests (Warburton *et al.,* 1993). Insecticides are the dominant class of pesticides used in most of the rice-growing countries. Plant protection chemicals currently cover about 30 per cent of total cultivated area in India, of which insecticides account for 61.39 per cent followed by fungicides (19.06%), herbicides (16.75%) and others (2.80%) (Shetty, 2004). In terms of total pesticide consumption, India is placed tenth in the world (Hundal *et al*., 2006). India has 170 mha of arable land with average pesticide consumption of 0.5 kg ha^{-1} (Shetty *et al*., 2008). Rice crop accounts for 22.80 per cent of total pesticide consumption in India (Dureja and Gupta, 2009).

Studies conducted in Philippines revealed that 55- 60 per cent of the pesticides used in rice were insecticides, 20-25 per cent fungicides and 5-16 per cent herbicides (Vander and Koeman, 1988). Moody (1990) reported that the use of herbicide was increasing rapidly all over the world due to the escalating cost and reduced availability of labour. The pesticide use had increased more rapidly in developing countries than in developed countries.

Rice cultivation in Kuttanadu, the "rice bowl of Kerala", is of an intensive nature compared to many other parts of the state. Majority of the farmers grow high-yielding varieties of rice, necessitating the use of high levels of chemical inputs (Alexander and Krishnakumari, 1990). Due to the high temperature and relative humidity prevailing, the area is prone to infestation by many pests which demand regular application of pesticides in the region. Outbreaks of different pests have been reported from Kuttanadu at times by different workers (Ambikadevi *et al.,* 1998; Nalinakumari *et al.,* 2002). The farmers in Kuttanadu resorted to excess use of agricultural inputs including pesticides in the desperate bid to save the crop grown. Devi (2009), revealed that farmers often spray hazardous pesticides at higher doses than recommended causing high risk to farmer health and environment.

2.1.1 Herbicide application in paddy

The rice fields and waterways in Kuttanadu are infested with different kinds of aquatic weeds. It was reported that the spread of weeds was rapid after the construction of Thaneermukkom barrage. Sasidharan *et al.* (1990) reported that *Echinochloa crusgalli* (L) was the predominant grass species while *Cyperus difformis* (L) and *Fimbristylis miliaceae* (Linnaeus) are the major sedges observed in Kuttanadu. The most commonly used weedicides in Kuttanadu are 2, 4-D (phernoxone), followed by Cyhalofop butyl (clincher) and Metsulfuron methyl and Chlorimuron ethyl (Almix) (Devi, 2009). 2, 4-D is a highly selective herbicide toxic to broad leaved plants but less harmful to grasses (Sharma, 2007). Chemically 2,4-D (2,4-dichlorophenoxy acetic acid) is an aryloxyalkanoic acid known also as a 'phenoxy herbicide', which includes MCPA, mecoprop, triclopyr and 2,4,5-T. These chemicals have complex mechanisms of action against weeds, resembling those of auxins (growth hormones). Once absorbed 2,4-D is translocated within the plant and accumulates at the growing points of roots and shoots where it inhibits growth. The phenoxy acid group of herbicides are probably one of the widest used herbicide chemcial classes. In US 2,4-D was the third most used pesticide in the early to mid 1990s, over 31,000 tonnes of 2,4-D was used annually. In UK it is among the top six herbicides used by UK local authorities, and it ranked seventh among herbicides used on grassland and fodder crops and twentieth among herbicides used in orchards in 1992. 2, 4-D is widely used in India, with an annual consumption of 1,300 tonnes in 1994-95. Sasidharan *et al.* (1990) reported that 2, 4-D was effectively used in controlling rice weeds of Kuttanadu.

The most important weed associated with rice, *Echinochloa* spp., is considered to be the major Gramineae weed, due to its wide distribution and competitive skill possessing similar bio chemical mechanisms as rice. Dimitrios *et al.* (2000) reported that cyhalofop-butyl at the rate of 0.2 kg per ha was effective in controlling barn yard grass (*E. crusgalli)* in rice. Santaella *et al.* (2006) reported that cyhalofop-butyl, 2-[4-(4-cyano-2-fluorophenoxy) phenoxy propanoic acid butyl ester (R), is an aryloxyphenoxypropionate (AOPP) herbicide for the post-emergence control of grasses in rice at application rates of 300 g ai ha^{-1}, mainly against almost all *Echinochloa* species.

Almix is a recently used herbicide of the sulfonylurea group for controlling the sedges and broad leaved weeds that suppress the growth of crops. Almix is a combined product of Metsulfuron Methyl 10 per cent and Chlorimuron Ethyl 10 per cent. The herbicide shows systemic action and works through both contact and residual soil activity, providing weed control for a longer period. It enters the plant body *via* contact through its leaves and from the soil through the roots. Sulfonylurea herbicides are very effective inhibitors of plant cell division. They inhibit acetolactate synthase (ALS), a key enzyme in the pathway of branched chain amino acids (leucine, isoleucine and valine) in plants.

2.1.2 Insecticide application in paddy

Nair (1978) reported that the major pests in the rice ecosystems of Kerala were *Scirpophaga incertulus* (Walker), *Leptocorisa acuta* (Thunberg), *Nilaparvata lugens* (Stal), *Orseolia oryzae* (Wood-Mason), *Cnaphalocrocis* *medinalis* (Guenee) and *Nymphula depunctalis* (Guenee). The important insecticides recommended against these pests were (KAU, 2007). Devi (2009) reported the use of organophosphates like acephate, phosmamidon, quinalphos, triazophos, malathion, methyl parathion, monocrotophos, dimethoate, carbamates like carbaryl, synthetic pyrethroids like lamda cyhalothrin and neonicotinoids like imidacloprid in rice ecosystem of Kuttanadu. Cartap hydrochloride, a synthetic analogue of nereis toxin is now widely used in Kuttanadu. The effective application of these pesticides on major pests of rice has been reported from many parts of the world.

Kushwaha (1995) reported that methyl parathion, monocrotophos, phosphamidon and endosulfan were effective in the control of *S. incertulas* 8.4-9.50 per cent dead hearts and 8.20-9.40 per cent white ear heads as compared to 22.64 and 25.79 per cent respectively with control. Singh and Sharma (1998) reported that three applications of cartap hydrochloride (at 1.0 kg a.i. ha^{-1}) at 20 days intervals significantly reduced the damage by *S. incertulas* and *C. medinalis*. Panda and Mishra (1998) observed that imidacloprid, monocrotophos, buprofezin and endosulfan were effective against the hopper pests of rice. Mishra *et al.* (1998) reported that ethofenprox, cartap hydrochloride, monocrotophos and phophamidon were effective in the control of rice leaf folder, (*C. medinalis)*. Acephate was found to be effective against leaf folder infestation in rice (Korat *et al.,* 1999). Cartap hydrochloride @1.5 kg ai ha^{-1} and carbofuran @ 28.9 q ha^{-1} controlled all the pests during the early stages of transplanted rice (Dash *et al.,* 2001). Comparative effect of granular

insecticides against green leaf hopper (*Nephotettix virescens* (Distant)) on paddy was studied by Gupta and Verma (2001). They reported that the systemic soil insecticide aldicarb effectively reduced the pest population by 85.90 per cent and consequently increased the yield of paddy to 41.29 q ha^{-1}. The per cent reduction of pest population for pesticide treatments over control were 79.03, 77.72, 74.83, 74.58, 70.35 and 69.41 for isofenphos, phorate, disulfoton, endosulfan, carbofuran, and fensulfothion respectively. Panda *et al*. (2002) identified carbofuran and ethofenprox as the most promising granular and sprayable compounds for controlling stem borer and BPH in rice. Combination of acephate 45 per cent + Cypermethrin 5 per cent at 500 g ai ha^{-1} was found to be effective against *L. vaicornis* Fabr (Dhingra *et al.*, 2003). Bio-efficacy of acephate towards hopper pests of rice was evaluated by Bhavani and Rao (2005) and found that imidacloprid and acephate followed by cartap hydrochloride gave the highest efficacy against plant hoppers. In an experiment in West Bengal, a total loss of 55.4 per cent was estimated due to lepidopteran (31.8 %) and non- lepidopteron pests (23.6%). The treatment using carbofuran granules and monocrotophos spray
recorded least incidence of stem bore (39.4 % dead heart an 4.4 % white ear 11
head) and green leaf hopper (11.8 and 12.4 per 10 hills) (Rath, 2005).

Application of cartap hydrochloride 5 days before pulling the rice seedling reduced the incidence of dead hearts (35.6 %) and white ears (28.4 %) caused by yellow stem borer, reduced leaf damage by 47.7 per cent and increased the grain yield compared to carbofuran applied as drench treatment

(Karthikeyan *et al*., 2007). Gupta *et al*. (2006) evaluated granular formulations of phorate and carbofuran (1000 g a.i ha^{-1}) and spray formulations of beta-cyfluthrin (12.5 g a.i ha^{-1}), thiacloprid (120 g a.i ha^{-1}), phosphamidon and monocrotophos (500 g a.i ha^{-1}each) against rice insect pests under field conditions. Infestation by stem borer (*S. incertulas*), white ear head, whorl maggot (*Hydrellia philippina* Fer.) and leaf folder (*C. medinalis*) was lowest (7.27, 5.60, 8.59 and 8.60%, respectively) in plots treated with thiacloprid.

Karthikeyan *et al*. (2008) reported that the new generation insecticide spinosad @ 54 g a.i ha^{-1} was effective against rice stem borer, gall midge, leaf folder and whorl maggot which caused 63 and 49 per cent reduction in dead hearts and white ears, respectively, 94 per cent reduction in leaf folder and 50 per cent reduction in gall midge infestation. Spinosad treatment also resulted in 14 per cent increase in rice yield.

2.1.3 Fungicide application in paddy

The introduction of high yielding, fertilizer responsive varieties led to the introduction of many new diseases in Kuttanadu. Non judicious application of nitrogeneous fertilizers coupled with the warm, humid weather conditions prevailing in Kuttanadu catalysed the development and spread of diseases. Sheath blight and sheath rot are the two common fungal diseases in Kuttanadu. Hexaconazole, carbendazim, propiconazole , mancozeb, ediphenphos and Kitazin are the important fungicides used in Kuttanadu (Devi, 2009). The effective use of these fungicides had been reported from other rice growing tracts in India and world. The two sprays of hexaconazole at 28.8 to

57.6 g a.i ha^{-1} at 55 and 70 days after sowing gave effective control of sheath blight. Sood and Kapoor (1997) reported that hexaconazole was effective against the blast disease in rice. Ali and Pathak (1997), reported that hinosan was effective in controlling the sheath blight in rice and found to be less inhibitory to *Trichoderma harzianum* Rifai. bio control agent of *Rhizoctonia solani* Kuehn. Hexaconazole, carbendazim and ediphenphos were found to be more effective than mancozeb for the control of sheath blight (Tiwari, 1997; Sudhakar *et al.*, 2005). Propiconazole and hexaconazole were effective in controlling the false smut of rice. Lore *et al*. (2007) compared the performance of different fungicides against multiple diseases of rice and found that propiconazole @ 0.10 per cent concentration was the most effective fungicide against the three major diseases in rice, where the lowest mean disease severity was 15.70 per cent for sheath blight, 6.70 per cent for sheath rot and 9 per cent for brownspot compared to 51.40 per cent 24.8 per cent and 48.5 per cent in untreated controls of respective diseases. The second best fungicide was carbendazim 0.10 per cent followed by hexaconazole 0.10 per cent

2.1.3.4 Behavior of pesticide molecules in rice ecosystem

Pesticide behavior in rice eco-system is very complex and determined by many variables and their interactions. The pesticide molecules after application begin to disappear from the target area either by physical movement by the action of air or water or by the action of degradation forces (Taylor and Spensor, 1990). Pesticide movements include adsorption/ desorption by soil, diffusion, volatilisation, percolation, runoff and adsorption by plants and animals.

Adsorption - desorption is a dynamic process in which the molecules are continually transferred between the bulk liquid and solid phase (Koskenen and Harper, 1990). Sethuraman *et al.* (1980) found sorption accounting for 40-95 % dissipation of applied pesticides. Diffusion is the process of fluid flow due to concentration gradient and takes place in both gaseous and liquid phases of the pesticides applied on the rice fields. In an experiment ^{14}C labelled phorate was mixed with soil prior to submergence 45 per cent residues were readily released from submerged soils into water during the first three days after flooding. Volatalisation is the process by which applied pesticides turned into vapour phase and subsequently lost into the atmosphere. Seiber *et al.* (1986) showed that the volatalisation was highest on the days of application and decreased rapidly. Percolation is the vertical downward movement of water due to gravitational force. Ramanand *et al.* (1988) found that percolation of carbofuran was high in unpuddled clayey soil than in puddle soil. Run off, the lateral movement of water on land surface was found to be mainly responsible for the transport of pesticide molecules from rice field to surrounding water bodies like lakes, rivers etc (Roger and Bhuhian, 1993).

2.2. Pesticide residues in abiotic and biotic components of rice ecosystem

2.2.1 Pesticides residues in soil

The degradation and persistence of pesticides in soil depend on the nature and properties of soil. The organic herbicides when applied were adsorbed more by highly acid clay soil than neutral soils.

2, 4-D , the most commonly used herbicide in Kuttanadu was reported to be having low soil sorption and high potential for leachability. The persistence of 2, 4-D in soil and water had been studied by several workers around the world. 2, 4-D persisted for 30 days in soil. The dissipation of 2,4-D appears to be dependent on oxidative microbial-mediated mineralization, photo degradation in water, and leaching. Data indicates that 2,4-D degrade rapidly in soils (half life = 6.2 days), degrades rapidly in aerobic aquatic environments (half life = 15 days), and is relatively persistent in anaerobic aquatic environments (half life ranges from 41 to 333 days). 2,4-D esters volatilize readily, particularly in conditions of high temperatures and low humidity. The studies on dissipation of cyhalofop butyl were conducted by applying at the rate of 300 g ai ha^{-1} to the rice plots without standing water and the rice plots were flooded 3 days after application. There were no detectable residues of cyhalofopbutyl in any soil or water sample at anytime. The only quantifiable residue in the soil was the diacid on day 7 at 0.01 mg/kg and there were trace levels (< 0.01 mg/kg) of the cyhalofop acid and diacid in the day 3 to 7 samples . Metsulfuron methyl and chlorimuron ethyl which are the constituents of almix when applied in the rice field left no residues during harvesting of the crop.

The persistence of pesticides in soil may vary according to the type of pesticides applied in the field. Read (1987) reported that aldicarb degradation was much slower in soil with pH lower than 5.6 than neutral soil. Gill and

Yeoh (1980) reported that carbaryl was more persistent in acid sulphate soil (pH 3.7) than in other acid soils (4.2 to 4.8).

In a leaching study by Tajeda *et al.* (1997) revealed that monocrotophos and endosulfan residues leached beyond the soil surface upto a depth of 175 cm in paddy soil. It was also observed that ^{14}C labeled monocrotophos reached up to a depth of 30 cm within 60 days in column experiment. ^{14}C labeled chlorpyriphos was found up to a depth of 20 cm on the sixth day after application. Adsorption of chlorpyriphos on soil was 91 per cent suggesting strong affinity to soil and hence leaching loss was reduced. Carbosulfan was converted to carbofuran in soil and remained for 30 days (Varca and Tajeda, 1998).They also found that DDT degraded rapidly when exposed in the open field with a half life of 31 days. DDT and its metabolite DDE dissipated faster in tropical soil than in temperate countries with half – lives of 235 and 161 days respectively. Pany *et al.* (2008) investigated the persistence of 3 levels of phorate at low (1.0 kg a.i. ha^{-1}), recommended (1.5 kg a.i. ha^{-1}) and double the recommended rate (3 kg a.i.ha^{-1}) in the rice fields. The rate of degradation of phorate with all the 3 levels in the soil seemed to follow a first order reaction. The half-lives (RL_{50}) were calculated as 10.27, 9.12 and 8.65 days for low, recommended and double the recommended dose, respectively, in the soil.

Soil samples from rice fields of the Kaithal region from Dehradun contained residues of Carbendazim in the range of 0.03-0.001 ppm (Arora *et al*. 2008).

2.2.2 Pesticides residues in water

Bhatt *et al.* (2004) reported that the residues of 2, 4-D in water can be detectable upto 35 days. Monitoring studies for pesticide concentrations in river systems in Japan detected several herbicides commonly used in paddy fields (Nakamura, 1993 ; Nagafuchi *et al.,* 1994). Rani *et al.* (2001) reported that the dissipation of triazophos in canal water was fast and independent of the initial concentration. Tanabe *et al.* (2001) conducted seasonal and spatial studies on pesticide residues in surface waters of the Shinano river in Japan. Among the total of 53 chemicals found, 22 were herbicides, 15 were insecticides, 11 were fungicides, and 5 were metabolites. The concentrations of chemicals found ranged from 3 ngL $^{-1}$ (bromobutide) to 8200 ng L^{-1} (isoprothiolane). They analyzed water samples from irrigation tanks and drinking water source from major paddy growing areas and areas of intensive pesticides use in Sri Lanka and found that out of 544 samples analysed eight were positive for either chlorpyriphos (0.22 to 0.542 ppm) or diazinon (0.012-0.15 ppm) and one sample contained dimethoate (0.014 ppm). Inao *et al.* (2008) conducted herbicide monitoring studies in two Brazilian rivers during the rice growing season. At least one herbicide was detected in 41 per cent of the water samples from the Vacacaí River and 33 per cent from the Vacacaí-Mirim River, the most frequent herbicide in both rivers being clomazone. Anasco *et al*. (2010) measured pesticide residues in five freshwater areas that are directly affected by rice paddy effluents in southern Japan to determine their maximum concentrations and temporal variations. Of the 14 target pesticides examined, a total of 11 were detected in all stations. Mefenacet, fenobucarb, and flutolanil were the three pesticides with the highest maximum concentrations and were also detected frequently. Analysis of temporal variations of pesticides showed that herbicides had relatively higher concentrations in the earlier stages of the rice planting season, while insecticides and fungicides had relatively higher concentrations at the later stages. Studies on the pesticide pollution in the Ebro river delta (Spain) showed that individual pesticides concentrations in water above 100 ng L^{-1} for about 50 per cent of the compounds (six triazines, four phenylureas, four organophosphorous, one anilide, two chloroacetanilides, one thiocarbamate and four acid herbicides) and total pesticides levels above 5 μg L^{-1} in the draining channels .The most ubiquitous compounds had been bentazone and MCPA and the highest levels had been observed for malathion (up to 5825 ng L^{-1}) and MCPA (up to 4197 ng L^{-1}).

2.2.3 Pesticides residues in fish and other aquatic organisms

Pinhero *et al.* (1988) reported that phorate was highly toxic to the aquatic organisms. Tejada *et al.* (1997) observed that toxicity to fish for the three insecticides tested was in the order cypermethrin > parathion-methyl > monocrotophos under laboratory conditions. But when these insecticides were applied to rice-fish culture they had no apparent effect on fish survival. Residues of parathion-methyl in fish accumulated only up to 4 days but were non detectable thereafter. A study on the toxicity of parathion-methyl, monocrotophos, and a mixture of fenobucarb and chlorpyrifos to fish and frogs after a simulated overflow of paddy water was conducted in Philippines (Calumpang *et al.,* 1997). Varca and Tajeda (1998) reported that when carbosulfan was applied in rice fields it was rapidly converted to carbofuran and the concentration in water was biomagnified upto 100 times in fish (*Tilapia* sp), with the highest residues in gut tissues, then in fillets and least in cranial tissue. Following treatment at 15 days after transplanting, exposure of fish (*Oreochromis mossambicus*) to parathion-methyl and monocrotophos in drainage canals 10, 25, 50 and 100 m from the point of application did not result in mortality, but exposure to Brodan- the mixture of fenobucarb and chlorpyrifos resulted in 100 per cent mortality, 10 m from the point of application and 6 h after application. No mortality was recorded following treatment 45 days after transplanting. At 15 days after transplanting, 100 per cent mortality was observed for frogs exposed to fenobucarb and chlorpyrifos, 90 per cent mortality for monocrotophos and 63 per cent for parathion-methyl at 25 m from the paddy field, immediately, and 1 h after application, respectively. The risks to aquatic organisms of 15 organophosphate pesticides (OPs) with various uses in Taiwan, including acephate, chlorpyrifos, diazinon, edifenphos, fenitrothion, fenthion, methamidophos, methyl parathion [parathion-methyl], monocrotophos, phenthoate, phorate, phosmet, temephos, terbufos, and trichlorfon, were assessed by Sun *et al.* (2002). Based on the applied concentrations, there were only 3 formulations of acephate (including 25% EC, 50% WP, and 75% SP), having low or slight toxicity to aquatic organisms, that were safe for use in rice fields. Among the 12 pesticides, only acephate with 75% SP had a low potential risk to aquatic organisms. High potential acute toxicity risks to aquatic organisms were recorded for chlorpyrifos with 22.5% EC, diazinon with 10% D, fenthion with 50% EC, and phenthoate with 50% EC.

Nakagome *et al.* (2006) reported that the herbicides oxyfluorfen, oxadiazon as well as the insecticides lambda-cyhalothrin, fipronil and carbofuran were found to be highest toxicity to *Daphnia magna* Straus. Samanta (2006) reported that DDT and its metabolites were the major constituents of the residues followed by HCH, heptachlor, endosufan and aldrin in fish samples collected from River Ganga in West Bengal. The total residues were in the range of 1.3 to 73.6 ng g^{-1} at Farakka, 7.9 to 90.4 ng g^{-1} in Barrackpore and 2.6 to 15.9 ng g^{-1} in Haldia (on fresh weight of fish) with observed mean values of 14.3, 37.9 and 7.6 ng g^{-1}. Klemick and Lichenberg (2008) reported the adverse effect of pesticides on fish population in rice eco-systems of Vietnam. Cock *et al.,* (2010) recorded reasonable coherence between pesticide concentration (in water and shellfish), toxicity, and mortality shellfish in the Ebro River Delta (Spain), for the different locations studied. Based on the toxicity data measured on samples, the main contributors to the total ecotoxicity were found to be malathion, and to a lesser extent diazinon and molinate.

2.2.4 Pesticide residues in rice plant, straw and grains.

Pesticides are applied to paddy field either as liquid or as solid. Formulations which are applied as sprays are directed mainly to foliage and absorbed and translocated by the leaves and the stem. Granular pesticides are applied to the soil and absorbed by the roots and translocated to other plant parts. In paddy, the grains and straw are harvested 35-45 days after the last application of pesticides. Hence the chance for contamination of rice grains with pesticides is meager. Tejada and Bajet (1990) found that of the seven pesticides formulations used, carbofuran and chlorpyriphos residue were translocated to rice grains and remained till harvest. Varca and Tajeda (1998) reported that cabosulfan remained as parent component in rice leaves upto 7 days.

Tejeda and Bajet (1990) reported that when monocrotophos, cypermethrin and parathion-methyl were applied to rice field, there were no residues on rice grains at harvest time. Phorate was detectable in rice grain and straw when applied at 1.25 and 1.5 kg ai ha^{-1} 40 days after sowing and at all doses (1.5-1.5 kg ai ha^{-1}) 60 days after sowing (Beevi and Visalakshi, 1992). Chlorpyriphos residues were detected in rice stem up to 5 days after spraying while the residues were detected upto fifteenth day in rice leaves. Monocrotophos residues were detected in rice leaves up to three days (Tejada *et al.,* 1993). The fate of ^{14}C labelled chlorpyriphos was studied by Tajeda *et al.* (1997). They reported that the pesticide residues were not detected in rice grains at the time of harvest when applied in rice field.

Tayaputch (1998) revealed that trace amounts of banned organochlorine insecticides could be found in milled and husked rice grain samples collected from different parts in Thailand. Residues of organophosphate and carbamate insecticides such as monocrotophos, malathion, carbofuran, isocarb and carbaryl were also detected in rice grain samples but at low levels.

In India monitoring studies conducted by All India Network Project on Pesticide Residues revealed that during the period 1985 to 1995 more than eighty per cent of the rice grains samples analyzed were found to be contaminated and the contaminants were predominantly HCH or DDT. Residues of HCH varied from traces to 5.32 ppm and DDT from 0.005 and 1.32 ppm (AICRP (PR), 1996). Later the contaminants were found to be carbofuran, phorate and endosulfan (AICRP (PR), 2003).

Ciscato *et al*. (2003) reported that out of 32 rice samples, pesticide residues were detected in 21.90 per cent of the analyzed samples. The pesticides detected were chlorpyrifos, fenitrothion, monocrotophos, pirimiphos-methyl and triadimefon.

Deka *et al.* (2004) reported the presence of pesticide residues in unpolished, polished and parboiled rice in Assam. Chen *et al*. (2007) investigated the residue levels of four hexachlorocyclohexane (HCH) isomers (α-HCH, β-HCH, γ-HCH and δ-HCH), 4,4’ dichloro-diphenyl-trichloroethane (DDT) congeners (p,p-DDE, o,p-DDT, p,p-DDD, and p,p-DDT), heptachlor, heptachlor epoxide, aldrin, dieldrin and endrin in rice and its bran. Concentrations of organochlorine pesticides (OCPs) for ∑HCH ranged from 0 to 0.039 mg kg^{-1} in the rice and 0 to 0.057 mg kg^{-1} in its bran. For ∑DDT, the concentrations ranged from 0 to 0.053 mg kg^{-1} in the rice and 0 to 0.051 mg kg^{-1} in its bran.

In an experiment conducted in China, pesticide residues were detected in rice grains and followed the order triazophos > acephate > methamidophos > chlorpyriphos > imidacloprid. The residues in straw, rice hull and grain were 46.0 per cent, 36.60 per cent and 17.40 per cent respectively (Qian *et al.*, 2008). Pany *et al.* (2008) reported that maximum uptake of phorate by rice plant was observed on 7th day after treatment. Arora *et al.* (2008) detected residues of carbendazim in rice grains at 0.001 mg kg^{-1} level. Hui *et al,* (2008) reported that residues of acephate in rice grains and straw when applied before heading were less than 0.1 mg kg^{-1}. Chen *et al*. (2007) investigated the occurrence of acetylcholinesterase (AChE)-inhibiting organophosphorus (OP) pesticide residues in milled rice samples obtained form local markets in China The results showed that 9.30 per cent of the samples contained detectable residues of at least one of the seven target OP pesticides (chlorpyrifos, dichlorvos, omethoate, methamidophos, parathion-methyl, parathion and triazophos) mainly used for agriculture in China, with concentrations ranging 0.011-1.756 mg kg^{-1}. They reported that pesticides residues in rice grains and straw as 0.18 µg g^{-1} and 0.01 µg g^{-1} when applied at 240 g ai ha^{-1} respectively. When applied at 500 g ai ha^{-1}, the residues were 0.106 µg g^{-1} and 0.03 µg g^{-1} respectively for rice grains and straw.

2.2.5 Pesticides residues in birds, mammals and other non-target organism

Tejada *et al.* (1993) reported that pesticide residues on test animals were below the maximum residue limit set by FAO/ WHO. In a ^{14}C labeled experiment rice fodder fortified with ^{14}C- Carbofuran was fed to lactating goats. The ^{14}C carbofuran was metabolized and excreted in urine (77 %) , faeces (3 5) and milk (0.05 %) (Tejada *et al.* 1997). Bioassay studies conducted by Sontakke *et al.* (2005) revealed that among the four insecticides tested, carbofuran was the most toxic to earthworm followed by quinalphos. Phorate and cartaphydrochloride were found to be safe to earth worms at their recommended dose. Monocrotophos is reported to be highly toxic to birds and it was implicated in a large number of bird death incidents affecting a wide variety of avian species (WHO, 2009).

2.2.6 Effect of pesticides on natural enemies in rice ecosystem

Application of synthetic pesticides for pest control adversely affect the natural enemy population of rice ecosystem. The population of mirid predator, *Cyrtorhinnus lividipennis* (Reuter) and spiders were significantly reduced in phorate treated rice fields in Kuttanadu (Beevi and Visalakshi, 1992). Relative toxicity of different insecticides to natural enemies of rice ecosystem was evaluated by Panda and Mishra (1998). A high degree of safety was observed in case of phosphamidon, cartap and diflubenzuron to spiders, deltamethrin to *C. lividipennis* and cartap to *Paederus fuscipes* Curtis. The influence of commonly used insecticides on predators of leaf and plant hoppers in rice was studied by Kumar and Veluswamy (2000) found that acephate, chlorpyriphos and monocroto*phos* were safe to spiders, *Lysocapseudo annulata, Tetragnatha javana* Thorell and *Paederus fuscipes* Curtis while acephate was also safe to *Microvelia douglasi atrolineatea*

Bergroth and *C. lividipennis*. Phorate and carbofuran were more toxic to both *M. atrolineatea* and *C. lividipennis.* Panda *et al.* (2002) observed that granular lindane and isazofos and sprayable monocrotophos, quinalphos 20 CS and carbaryl were highly toxic for spiders in rice eco system. Patel *et al.* (2004) evaluated the effectiveness of the effect of insecticides commonly used for the control of rice insect pests on spiders. All the insecticides (profenophos+ cypermethrin, dichlorvos, endosulfan, azadirachtin , acephate, cartap, carbofuran, imidacloprid, and fenucarb) significantly reduced spider population than the control. Azadirachtin recorded the highest spider population (0.39 per plant) followed by carbofuran (0.36 per plant) and endosulfan (0.34 per plant). Bhavani and Rao (2005) reported that imidacloprid ranked first in terms of safety to spiders followed by acephate while acephate ranked first in terms safety to mirid bugs followed by imidacloprid and cartap hydrochloride. The effect of some insecticides was studied on the population of predatory spiders, beetles, carabid beetles, dragon flies and damsel flies by Sreenivas and Madhumathi (2005). Granular formulations of cartap hydrochloride, chlorpyriphos, fipronil, carbofuran and sunny neem at 5 ml per litre were found to cause 20-50 per cent reduction in population while BPMC (feobucarb) and phenthoate were highly toxic to spiders, damsel flies and dragon flies with more than 70 per cent reduction in population over the control. Ramudu and Misra (2005) studied the residual effect of new insecticides and combinations against *Leptocorisa* spp and found that clothianidin and combination products involving acetamiprid with quinalphos / chlorpyriphos and monocrotophos rcorded significantly lowest bug population.

Among the new generation insecticides viz., imidacloprid, cartap hydrochloride, ethofenprox, fipronil, thiodicarband spinosad, fipronil and ethofenprox were recorded to be safe to spider and mirid bug population in rice ecosystem (Sethuramu *et al*., 2006). In an experiment conducted at Pattambi, it was observed that no significant reduction in population of predators like damsel flies (*Agriocnemis* sp), green mirid bugs (*C. lividipennis* and larval parasites (*Stenobracon* sp.) was recorded in plots treated with cartap hydrochloride applied 5 days before pulling the rice seedlings (Karthikeyan *et al.,* 2007). Karthikeyan *et al.* (2008) reported that the new generation insecticide spinosad caused no significant effect on spider population and was safe to spiders that predominate the predatory fauna in rice while monocrotophos and lambdacyhalothrin significantly reduced the spider and larval parasitoid populations in the rice ecosystem.

2.3 Impact of Pesticide on farmer health

In Kuttanadu area, known, the pesticide use was reported to be very high and there were several mass media reports on its impacts on the ecosystem (Devi, 2010). But the scientific efforts to quantify these externalities are only a few. Dinham, (1993) reported frequent cases of cancer of the lip, stomach, skin and brain, lymphoma, leukemia and multiple myloma from the Kuttanadu linking the same to high pesticide use in the area. Rakhesh, (1999) studied the externalities associated with pesticides in

Kuttanadu, and found that pesticide poisoning leads to both explicit and implicit costs for the applicator/ farmer. Krishna (2001) reported that among the health hazards induced by pesticides, the skin allergy and headache were most prominent in Kuttanadu. The micro level study conducted by Devi (2009) (the SANDEE-funded project) showed 73 cases of hospitalisation due to occupational exposure among a sample of 1,135 spray events in Kuttanadu area alone during the summer rice season. The average expected health costs to the pesticide applicators in this case was estimated to Rs 38 per spray event.

Surveys conducted by Aponso *et al.* (2003) in SriLanka revealed that farmers take minimal precautions when handling and 7 per cent of farmers do not apply recommended dosage. Approximately 21 per cent farmers suffered from acute toxicity symptom such as dysuria, myalgia and headache. Lung cancer risk was 3-fold higher for those with > 109 days of lifetime exposure to carbofuran compared with those with < 9 lifetime exposure days, with a significant dose-response trend for both days of use per year and total years of use.

2.4 Risk assessment and management of pesticide residues

Inao *et al.* (2008) suggested that increasing water holding period would significantly reduce the herbicide concentrations in runoff water. They suggested that river water contamination by rice herbicides is probably caused by the rice water management used in the fields. The maintenance of flooded areas makes herbicides prone to contaminate the environment. To reduce the environmental contamination risk it is necessary to adopt measures to avoid overflow of flooded rice fields, keeping paddy water in the field for time enough to reduce the herbicide concentration before its release and enhancing the quality of the levees to reduce the probability of paddy rice overflow. According to Watanabe *et al*. (2007) an extension of water holding period to 10 days after herbicide application from the currently recommended period of 3-4 days was found to be a good agricultural practice for controlling pesticide run-off from paddy fields. They recommended an intermittent irrigation scheme using a automatic irrigation system with high a drainage gate for use during the water holding period for reducing pesticide runoff from paddy fields. Inao *et al.* (2008) applied the PADDY and PCPF models to controlling pesticide runoff losses from paddy fields and to ecological risk assessment in the aquatic environment. The recommendation from model simulations for reducing pesticide runoff from paddy fields are 1) application of an intermittent irrigation scheme with a high drainage gate and 2) application of a longer water holding period after pesticide application.

MATERIALS AND METHODS

3. MATERIALS AND METHODS

All the experiments connected with the present study were carried out in the different sites of Kuttanadu during 2006-2009. The samples for the estimation of pesticide residues collected from the experimental fields were brought to the All India Network Project on Pesticide Residue Laboratory, College of Agriculture, Vellayani for further processing and estimation of residues.

3.1 SURVEY

A detailed survey was conducted to study the consumption and use pattern of pesticides in the major rice growing tracts of Kuttanadu during 2007-08 and 2008-09. The catchment area of three major rivers in Kuttanadu, *viz*., Pampa, Manimala and Meenachil were selected for the study. From each catchment four different rice growing belts were selected. In Pampa catchment, Neerattupuram, Edathua, Thakazhy and Karumady were selected. Kidangara, Ramankari, Nedumudi and Pallathuruthy were selected from the catchment of Manimala. In catchment of Meenachil, Thiruvarpu, Kumarakom,Vechoor and Vaikom were selected.

The time frame of the study for catchment of Pampa and catchment of Manimala was from October-November to January – February, 2009 which is the main crop season in Kuttanadu while for catchment of Meenachil, the period was from June to September,2009 representing the main season of the locality.

Data on pesticide consumption in Kuttanadu were collected by interviewing rice farmers who were cultivating rice for the past ten years. Ten farmers were randomly selected from each location and thus forty farmers were surveyed to represent each catchment. A suitable questionnaire was prepared and used for collecting the data. The frequency of pesticide application was recorded during the main crop season of Puncha for two years, 2007-08 and 2008-09. To have a comparison of the present pesticide use pattern with the one previously prevailed in the region, data on pesticide application followed by the same study group five years before (2002-03) were also collected. One padasekharam (collective farm) each from the four different rice growing belts in the three selected river catchments of Kuttanadu were chosen for studying the average pesticide consumption per hectare.

Data on pesticide use pattern was collected from ten selected farmers in each rice growing belt. Farmers were interviewed to collect required information which included type of pesticides used, rate, time, frequency and method of application, equipments used, stage of crop, against which pest/ disease used, whether recommended or not etc. Information on different practices followed by the farmers regarding pesticide use and storage were recorded. The data was utilized to explore the reasons for not following GAP in pesticide use and storage.

Five spray men were selected in each locality and interviewed to find out the direct health impacts of repeated pesticide exposure such as headache, dizziness, vomiting, unconsciousness, stomach pain, weakness etc.

3.2 Validation of Multi Residue Methods (MRM) for pesticide residue analysis of water, soil, rice grain and paddy straw, fish and duck meat

Multi Residue Methods for each substrate were validated using the validated protocol. The following glass wares, reagents and equipments were used for the study.

Laboratory glass wares

1. Microseparator, one litre brown bottle (supplied by FAO)

2. Separatory funnel 500 ml, 1 L

3. Sintered chromatographic glass column 2.2 cm x 60 cm

4. Microsyringe 10µl, 500 µl

5. Round bottom vacuum flask 500 ml

6. Conical flask 250 ml, Beaker 100, 250, 500 ml, Funnel 75 mm dm, graduated test tubes.

Chemical reagents

1. Acetone AR grade

2. Acetonitrile AR grade

3. Dichloromethane AR grade

4. n-Hexane HPLC grade

5. Hexane AR grade

6. Methanol AR grade

7. Sodium Sulphate AR grade (anhydrous)

8. Sodium Chloride AR grade

9. Alumina

10. Silica Gel

11. Florisil AR grade

12. Petroleum ether

Equipments

1. Electronic weighing balance (Sartorius)
2. Mechanical shaker
3. Rotary vacuum flash evaporator
4. Hot air oven
5. Magnetic stirrer
6. Solid phase extraction unit SPE Manifold Biotage Sweden AB
7. High Performance Liquid Chromatograph (Shimadzu LC 20AT)
8. Gas Chromatograph– (Shimadzu GC 2010 A)Gas Chromatograph – Mass Specttrometry (ShimadzuGC/MS 2010QP Plus)

All the glasswares were washed in clean water, then rinsed with distilled acetone thrice and dried at 100°C for 30 minutes. Syringes were thoroughly rinsed with acetone followed by hexane. Solvents used in the study were glass distilled before use. Sodium sulphate was prewashed with acetone, dried at room temperature and then activated in an oven at 110 °C for three hours.

Preparation of Standard Stock Solution for preliminary recovery studies

Certified reference material of lindane, alpha endosulfan, methyl parathion, chlorpyriphos, and ethion having purity ranging from 96 to 99.00 per cent from M/s Sigma Aldrich supplied through Project Coordinating Cell, All India Network Project on Pesticide Residues, IARI, New Delhi were used to prepare stock solutions of individual pesticide using acetone. Aliquots of stock solution of individual pesticides were drawn in a separate volumetric flask so as to get a final mixture of five pesticides at concentration level of 50 mg L^{-1}. Final volume was made up with n-hexane and lower concentrations (10, 5,2.5 and 1 ppm) were prepared by serial dilution.

Preparation of Standard Stock Solution for validation of Multi Residue Method

The MRM which was found to be the most efficient in the preliminary trial was selected for validation studies using 11 pesticides

(two organochlorine, six organophosphorous, one synthetic pyrethroid and one fungicide) representing all the major groups of pesticides prevalently used in Kuttanadu. Analytical grade standards of pesticides *viz*. lindane, alpha endosulfan, phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, ediphenphos, lambda-cyhalothrin, deltamethrin and hexaconazole having purity ranging from 96 to 99.00 per cent from M/s Sigma Aldrich supplied through Project Coordinating Cell, All India Network Project on Pesticide Residues, IARI New Delhi were used to prepare stock solutions of individual pesticide using acetone. Aliquots of stock solution of individual pesticides were drawn in a separate volumetric flask so as to get a final mixture of eleven pesticides at concentration level of 50 mg L^{-1}. Final volume was made up with n-hexane and lower concentrations (10, 5, 2.5 and 1 mg L^{-1}) were prepared by serial dilution.

Standardization of GC condition

Gas Chromatograph – (Shimadzu GC 2010 A) with ECD ^{63}Ni with electron capture detector fitted with DB-1 column (dimethyl polysloxane, 30m X 0.25mm, 0.5µm film thickness) was used for analysis. Ultra High Purity (99.999 %) nitrogen was used as carrier gas with flow rate 1.5 ml min^{-1} linear velocity 39.90 cm sec^{-1}. The temperature at injection port and detector port were kept at 250° and 300°C respectively. An oven temperature programme (170-220 °C ; 3 °C per minute;2 minutes holdtime; 220-270 °C; 5 °C per ; 10 minutes hold time) was developed to get proper separation of all pesticides used in the analysis.

Gas Chromatograph – (Shimadzu GC 2010 A) with Flame thermionic detector fitted with DB-5 column (30m X 0.25mm, 0.25µm film thickness) was used for analysis. Ultra High Purity (99.999 %) nitrogen was used as carrier gas with flow rate 1.3 ml min^{-1} linear velocity 34.90 cm sec^{-1}. The temperature at injection port and detector port were kept at 250° and 290°C respectively. The make flow (Nitrogen) was 20.00 ml min^{-1} .Hydrogen and air flow were 3.00 ml min^{-1} and 140 ml min^{-1} repectively. An oven temperature programme (170-220 °C ; 3 °C per minute;2 minutes holdtime; 220-270 °C; 5 °C per ; 10 minutes hold time) was developed to get proper separation of all pesticides used in the analysis.

Determination of Limit of Detection (LOD)

Working standards of 1, 0.5, 0.4, 0.3, 0.2 and 0.1 mg L^{-1}were prepared. One micro litre of each concentration was injected in GC under set standard GC conditions. Each standard was injected in three replications. The limit of detection of instrument for each pesticide was calculated based on the lowest quantity of pesticide standard that can be identified under standard GC conditions. Lowest concentration for which a response of >3 times the noise peak obtained was considered as LOD of the particular compound. The linearity response line (calibration curve) was plotted with quantity of pesticide at X-axis, and peak area counts at Y axis.

Standardization of HPLC condition

High Performance Liquid chromatography – Shimadzu LC 20 AT, Photodiode Array Detector (PDA) fitted C-18 (Phenomenex) reverse phase column was used for analysis. The mobile phase was acetonitrile: water (80: 20) with flow rate of 1ml min^{-1}. Data was collected by PDA detection from 269 to 280 nm.

3.2.1 Validation of Multi Residue Methods (MRM) for water

Preliminary recovery studies were conducted using candidate pesticides to represent different groups of pesticides following three different methods for estimation of multiresidues in water.

3.2.1.1 Method I (Hernandez *et al.*, 1993)

Water was collected from organically grown rice fields and fortified at 1ppb level with a mixture of five pesticides prepared in acetone. Water (1L) was fortified with standard mixture so as to get a fortification level of 1 µg

L^{-1}.The fortified water sample (0.5 L) was transferred to one litre separatory funnel to which 100g sodium chloride was added and shaken till completely dissolved. The residues were extracted thrice with dichloromethane (100:50:50 ml) each time shaking vigorously for one minute. Lower organic layer was collected by passing through 5 g anhydrous sodium sulphate supported on washed glass wool in 4 inch filter funnel. The organic layers were combined and concentrated to 0.5 ml using rotary vacuum evaporator. Concentration step was repeated thrice in presence of n- hexane to remove all traces of dichloromethane. The final volume of the extract was made up to 0.5 ml resulting in a concentration factor of 1000. The extract was analyzed using GC with ECD detector.

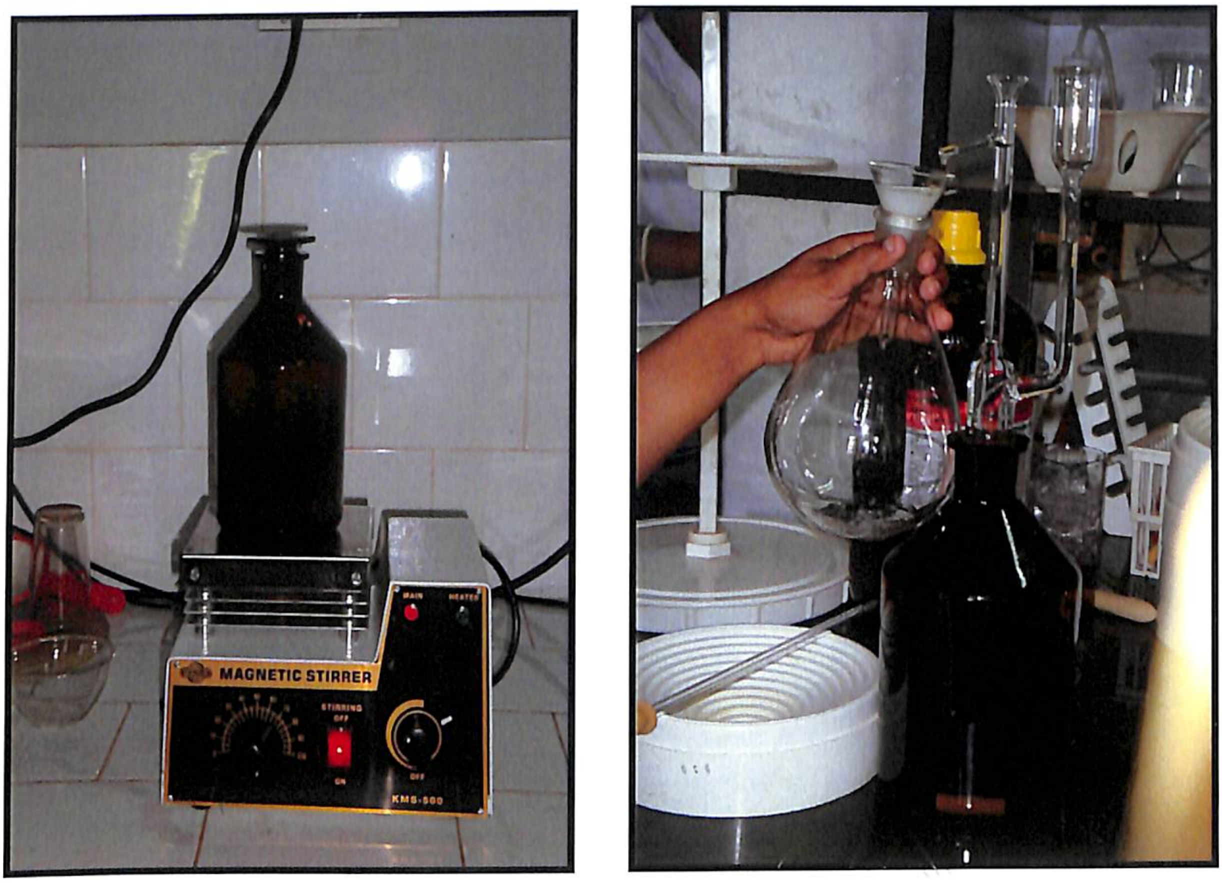
Analysis of 2,4-D

Water (1L) was fortified with 2,4-D so as to get a fortification level of 0.05 , 0.1 and 0.2 mg L^{-1}.

For analysing phenoxy herbicide 2,4-D, 10 ml concentrated sulphuric acid was added to the aqueous phase left in the above extraction and The residues were extracted with dichloromethane (100:50:50 ml) as mentioned above and the final volume was made up to 0.5 ml using actetonitrile and analyzed using HPLC.

3.2.1.2 Micro separator method

Two litres of water sample fortified with a mixture of five pesticides so as to get a fortification level of 1 µg L^{-1}. The water sample (one litre) was taken in one litre brown bottle supplied by International Atomic Energy Agency Vienna (Plate 1) to which 20g NaCl and 20 ml n-hexane were added and shaken vigorously for 10 minutes using magnetic stirrer. Then it was allowed to rest for 5



A. Sample processing B. Extract collected using micro separator

Plate 1. Micro separator method of pesticide residue analysis in water.

minutes. Then micro separator was inserted in the separatory bottle. Distilled water (130-140 ml) was slowly added through the inlet of the micro separator. The n-hexane layer was collected into evaporating flask. Last drops were collected after passing through anhydrous Na_2SO_4 and n- hexane was concentrated to 0. 5 ml using a rotary vacuum flash evaporator. The extract was analyzed using GC with ECD

3.2.1.3 Solid phase extraction method

The extraction was carried out using the solid phase extraction unit SPE Manifold Biotage Sweden AB. Isolute C-18 cartridges (EC) were used in the SPE manifold. The cartridge was conditioned by passing one ml of Methanol / Acetonitrile mixture (1:1) three times followed by 2 ml of water sample for three times. Then the catridge was loaded with one litre of water spiked with the pesticides at 1ppb level at the rate of 6ml per minute.The pesticides were eluted sequentially thrice with 1 ml of Methanol / Acetonitrile mixture (1:1) and concentrated using rotary vacuum flash evaporator and solvent exchange to n-hexane is done.The extract was analyzed using GC with ECD.

3.2.1.4. Validation of MRM involving additional candidate pesticides.

Based on the performance of the three methods mentioned above, the best method was selected for further trial on validation using additional candidate pesticides at different concentrations following the method mentioned in item 3.2.1.1. The pesticides selected were based on the findings of the survey conducted in Kuttandu rice ecosystem. The pesticides selected included the most widely used ones *viz.* phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, lindane, alpha endosulfan, lambda-cyhalothrin, deltamethrin, hexaconazole and ediphenphos. The water sample (1L) was fortified with the standard mixture so as to get 100, 50,10 and 1 µg L^{-1} .

In order to validate pesticide residue analytical method recovery studies were carried out at specified fortification levels. Five important validation parameters *viz.,* recovery percentage, repeatability, reproducibility, linearity and limits of detection and quantification (Zanella *et al.,* 2000) were evaluated for the pesticides at the laboratory conditions at AINP on Pesticide Residues, College of Agriculture, Vellayani. The repeatability of the selected analytical method was determined by repeating the method at different fortification levels. The reproducibility of method was evaluated by analyzing the fortified samples over three consecutive days.

3.2.2 Validation of Pesticide residue analytical methods for soil

Preliminary recovery studies were conducted using candidate pesticides to represent different groups of pesticides following two different methods for estimation of multiresidues in soil.

3.2.2.1 Dry soil method (Sharma, 2007)

Soil samples were collected from organically grown rice fields using box cores measuring 5cm X 5cm X 5cm. One core of soil sample (approximately 625 cubic cm) was drawn from subplots and pooled and sampled and dried in shade. The soil was fortified at 0.1 ppm level with five pesticides viz., lindane, alpha endosulfan, methyl parathion, chlorpyriphos, and ethion. 100 g soil was weighed and added a few drops of liquid ammonia and mixed well and left for half an hour till ammonia got evaporated completely. The contents were transferred into Soxhlet apparatus and refluxed using hexane : acetone (1:1 v/v) for 6-8 hours. The extract was concentrated and the final volume was made up to 10ml, using 10 per cent acetone in hexane. The extract was further cleaned up using anhydrous alumina and sodium sulphate column. The extract was analyzed using GC.

3.2.2.2 Wet soil method

The sample (50 g) was shaken in a platform shaker at 60 rpm and kept for one hour. The contents were decanted through Whatman No.40 filter paper. The extraction was repeated for two times. The combined extract was concentrated to nearly 75 ml and partitioned using DCM thrice with 100, 50, 50 ml respectively. This was concentrated and made up the volume to nearly 5ml. Column clean-up was done using activated acidic alumina and celite. The elulant was concentrated to 5 ml in n-hexane-acetone mixture and analysed in GC.

Analysis of 2,4-D in soil

Fifty gram of representative soil was weighed and fortified with 2,4-D at 0.2, 0.1 and 0.05ppm levels and was kept for one hour and then added 0.5ml of acetic acid. 100 ml acetone was then added and shaken well and kept for one hour and decanted through Whatman no.40 filter paper. The extraction was repeated twice and the extract was concentrated to nearly 75 ml. Then the concentrate was partitioned using dichloromethane thrice with 100, 50, 50 ml of DCM each time. This was concentrated and the volume made upto nearly 5ml. The extract was made up in acetonitrile and analyzed using HPLC with photodiode array detector. The repeatability and reproducibility was proved at these levels.

3.2.2.3. Validation of MRM involving additional candidate pesticides

Based on the performance of the two methods mentioned above, the best method was selected for further trial on validation using additional number of pesticides at different concentrations following the method mentioned in item 3.2.3.2. The pesticides selected were based on the findings of the survey conducted in the Kuttandu rice ecosystem. The pesticides selected includes, phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, lindane, alphaendosulfan, hexaconazole, ediphenphos, lambda cyhalothrin and deltamethrin at concentrations.

3.2.3. Pesticide residue analysis in paddy grains (Sharma, 2007)

Representative rice grain samples (50g) were collected from organically grown rice field and were pooled. The samples were fortified with selected pesticides viz., phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, lindane, alpha endosulfan, hexaconazole, ediphenphos, lambda cyhalothrin and deltamethrin at concentrations of 0.2, 0.1 and 0.05 mg kg^{-1}. After keeping for one hour the grain samples (15 g) were taken and ground and blended with 350 ml acetone/water 65:35 for 2 minutes at high speed. The extract was filtered and transferred to 1 litre separatory funnel and the residues were extracted with 200 ml mixture of hexane : dichloromethane (1: 1) by shaking vigorously for 1 minute. The lower aqueous layer was transferred to another 1 litre separatory funnel. The organic phase of first separatory funnel was dried by passing through approximately 5 g sodium sulphate supported on pre washed cotton in 4" filter funnel. Saturated sodium chloride solution (10 ml) was added to the separatory funnel containing aqueous phase and shaken vigorously for 30 seconds. This was then extracted twice with 100 ml of dichlormethane each. The extracts were pooled and concentrated. Concentration step was repeated in presence of hexane to remove all traces of dichloromethane and final volume made upto 5ml in n-hexane. One ml of above solution was diluted to 10 ml with 10 per cent acetone in hexane. The chromatographic column was packed with 4 g activated florosil, followed by 2 g of sodium sulphate. The column was eluted at about 5ml/min with 50 ml elutant (50 % dichloromethane : 1.5 % acetonitrile :48.5 % hexane v/v/v). The elutant was centrifuged to 1 ml. The extract was analyzed in GC-ECD.

3.2.4 Pesticide residue analysis in paddy straw

Paddy straw samples (50g) were collected from organically grown rice field powdered, pooled and fortified with selected pesticides *viz*., phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, lindane, alpha endosulfan, hexaconazole, ediphenphos, lambda cyhalothrin and deltamethrin at concentrations of 0.2,0.1 and 0.05 mg kg^{-1} and kept for one hour. The samples (5g) were then ground to which added 100 ml dichloromethane and shaken well. The extract was filtered and extracted by two times with 50 ml dichloro methane each. The filtrate was combined and concentrated to nearly 5 ml and passed through a chromatographic column packed with 10g alumina in between layers of anhydrous Na_2SO_4 . The elutant was evaporated in rotary vacuum flash evaporator and finally made up to 5 ml in n-hexane. The extract was used for analysis in GC-ECD.

3.2.5 Pesticide residue analysis in fish (Sharma, 2007)

Fish samples were collected from fresh waterbodies in Kuttanadu. The edible portion of samples were cut into pieces, macerated and pooled. 25 g of sample was weighed and fortified with selected pesticides *viz.*, phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, lindane, alpha endosulfan, hexaconazole, ediphenphos, lambda-cyhalothrin and deltamethrin at concentrations of 0.2, 0.1 and 0.05 mg kg^{-1} and kept for one hour. Then the sample was ground thoroughly in a mortar and added 100g anhydrous sodium sulphate and mixed well. The mixture was taken in a 500 ml conical flask and 150 ml petroleum ether was added and shaken well for 15 minutes in a shaker. The petroleum ether was decanted through anhydrous sodium sulphate placed in a glass funnel plugged with glasswool and collected in another beaker/conical flask. The residues were re extracted with two 100ml portions of petroleum ether by shaking 15 minutes each time in a shaker. The extracts were combined together and concentrated to 5 ml in vacuum flash evaporator. The extract was quantitatively transferred to a 100 ml separating funnel. To this 15 ml of acetonitrile saturated with petroleum ether was added and shaken well and allowed to separate. The bottom layer containing pesticide was transferred into one litre separating funnel containing 600 ml of water, 100 ml petroleum ether and 40 ml saturated sodium chloride solution. Extraction with acetone was repeated two more times and the bottom layer was poured to a one litre separating funnel, shaken well and allowed to separate. The aqueous layers were discarded and the petroleum ether layers from the two were combined, dried with anhydrous sodium sulphate and concentrated in a vacuum flash evaporator. Clean up was done using chromatographic column placed with 5 g anhydrous sodium sulphate followed by 25g activated florisil and another 10g sodium sulphate over it. The column was first eluted with 200ml petroleum ether containing 6 per cent ether. The residues were re-constituted in 5 ml petroleum ether for injection into gas chromatograph (GC-ECD) for estimation of residues.

3.2.6 Pesticide residue analysis in duck meat (Sharma, 2007)

Duck meat samples were collected from uncropped area of Kuttanadu and the edible portion was cut into pieces. A 20g macerated duck meat tissue was fortified with selected pesticides viz., phorate, dimethoate, methyl parathion, malathion, chlorpyriphos, lindane, alpha endosulfan, hexaconazole, ediphenphos, lambda -cyhalothrin and deltamethrin at concentrations of 0.2,0.1 and 0.05 mg kg^{-1} and kept for one hour. The sample was ground thoroughly and Sodium sulphate (40g) was added to the sample. The contents were mixed well using a stirring rod and allowed to stand for 20minutes and mixed again. 100ml hexane was added to the sample and blended for 1-2 minutes. The contents were filtered through glass wool. The hexane layer was extracted by passing through the bed of anhydrous sodium sulphate. The sample was again mixed with a stirring rod and 100ml hexane was added and extracted. The extraction was repeated with 70ml hexane, combining all three extracts in the same flask. The extract was concentrated and cleaned up and analyzed in GC-ECD. The same procedure was followed for animal meat (beef) also.

3.2.7 Recovery experiments on MRM for eggs

Recovery experiments were conducted for MRM for duck eggs as per the protocol (Sharma, 2007).Duck egg samples were collected from uncropped areas of Kuttanadu. Egg samples were broken and the contents were pooled. The sample (25 g) was then fortified with selected pesticides viz., phorate, dimethoate, methylparathion, malathion, chlorpyriphos, lindane, alphaendosulfan, hexaconazole,ediphenphos,lambdacyhalothrin and deltamethrin at 0.2 ppm level and kept for one hour. Sodium oxalate (2g) and 100 ml ethyl alcohol were then added and blended for 2-3 minutes. The extract was then filtered and transferred to separatory funnel. Diethyl ether (50 ml) was added and shaken vigorously for one minute. The upper organic layer was collected into another separatory funnel. Rextracted the aqueous phase twice with 50 ml mixture of diethyl ether and hexane (1:1) v/v). Combined all the three extracts and 500 ml of distilled water and 30 ml saturated sodium chloride solution were added. Shaken well for one minute. Water was drained and discarded. The organic layer was drained by passing through anhydrous sodium sulphate layer. The aliquot representing three gram of fat was cleaned up and analyzed in GC-ECD.

3.2.8 Recovery experiments on acephate, quinalphos and triazophos.

Recovery experiments were conducted to assess the recovery per cent of acephate, quinalphos and triazophos using the MRMs for water, soil, paddy grains, paddy straw, fish, duck meat and duck eggs as mentioned in 3.2. The extracts were analysed using GC-FTD.

3.3 Monitoring of pesticide residues in biotic and abiotic components of Kuttanadu ecosystem

The samples of water, soil, paddy grains, paddy straw, fish,mollusk, duck meat,eggs and animal meat (beef) were collected during the puncha season of 2007-2008 from the three catchments under study and were analyzed for the presence of pesticide residues. In each catchment, two padasekharams were selected for the collection of samples for monitoring of pesticide residues in the biotic and abiotic components. Samples of water and soil/sediment from the field and the point of discharge from paddy fields in each padasekharam were collected for estimation of residue at four growth stages of rice crop viz., seedling, tillering, booting and milky stage. Further, samples of soil and water from the drainage channels, stream and river bodies were also collected for estimation of residues (Plate 2). The samples were analyzed as per the protocol described in 3.2.

A. Rice field

B. Field outlet

C. Drainage channels

D. Streams

E. Reservoirs

Plate 2. Sampling sites in rice field

Plate 3. Duck meat for consumption from paddy ecosystem

Plate 4 Cattle fed with weeds from paddy ecosystem

Samples of paddy grain and straw at harvest were collected from the selected padasekharams and the pesticide residues were estimated following the methods as described in 3.2.

Samples of fish, mollusk, duck meat, eggs and beef were collected during cropped season (Plate 3). The samples were analyzed for the presence of pesticide residues as per protocol described in 3.2.

Pesticides were analyzed using GC and HPLC. The pesticides detected in GC were confirmed by GC-MS.

3.4 Impact of conventional pest control and IPM practices on major pests, natural enemies and neutrals

The study the impact of conventional and IPM practices on major pests, natural enemies and neutrals, two padashekarams were selected from the catchment of Manimala, one following IPM practices and the other following conventional pest control methods.

3.4.1 Assessment of population of pests and natural enemies

The population of pests, natural enemies and neutrals in the experimental field following IPM practices and conventional methods were assessed with a sweep net adopting the method of Reissig *et al.* (1986). The insects and pests collected were transferred to a polythene bag. A long cotton strip moistened with chloroform was introduced into the polythene bag without touching the sides and placed at the open end of polythene bag. After 10 minutes the dead insects were transferred from the polythene bag to a white blotting sheet and sorted. The number of pests, natural enemies and neutrals were counted. The insects were then identified based on the taxonomic characters.

3.4.2 Assessment of pest damage

Percentage incidence of stem borer was recorded following standard techniques. Ten numbers of hills were randomly selected and marked for recording observations. The number of infested hills, damaged tillers and the total number of tillers in the damaged hills were recorded from these ten randomly selected hills. Then the percentage incidence (PI) was evaluated as

$$PI = \frac{\text{No.of damaged hills in the sample X No. of damaged tillers X 100}}{\text{Total no. of hills X Total no. of tillers in the infested hills}}$$

The incidence of rice leaf roller was assessed in terms of total infested leaves in the plots. The total number of leaves (both damaged and undamaged) were recorded from the randomly selected ten hills in the plot. Depending on the extent of damage the leaves were graded visually into 3 categories and percentage damage (PD) was evaluated following the formula,

$$PD = \frac{(a\ X1) + (b\ X2) + (c\ X3)}{N\ X\ 3}$$

a= no. of leaves with slight damage (25 %)

b= no. of leaves with moderate damage (26-50 %)

c=no. of leaves with severe damage (above 51 %)

N= Total no of leaves (both infested and uninfested) from ten hills

3.4.3 Yield attributes of paddy from IPM and conventional field

The yield from the plots was recorded following crop cutting method. A square plot of 5m X 5m was demarcated and grain and straw yield were recorded separately. The yield is expressed as Kg ha^{-1} was computed by using the formula,

3.5 Field experiment to study the dissipation of commonly used pesticides in Kuttanadu rice ecosystem

Observations on the dissipation of commonly used pesticides in Kuttanadu were recorded from two padashekarams in Edathua which received the following treatment combinations. All the management practices except the plant protection were followed as per the recommended package of practices of Kerala Agricultural University (KAU, 2007).

3.5.1 Field situation- I

The plant protection practices followed by rice farmers in Kuttanadu in padasekharam I.

Variety : Uma

No.	Pesticide	Stage of application	Dose
1	2,4-D	Seedling stage 17 DAS	1 kg ha^{-1}
2	Chlorpyriphos	Early tillering 30DAS	0.25 kg ai ha^{-1}
3	Acephate	Late tillering 50DAS	0.45 kg ai ha^{-1}
4	Hexaconazole	Late tillering 50DAS	0.05 kg ai ha^{-1}
5	Lambda cyhalothrin	Flowering stage 90 DAS	0.125 kg ai ha^{-1}

3.5.1.1 Dissipation studies of pesticides

For experimental purpose, the area was divided into plots of 2m X 30 m. The plots were sub divided into small plots of 2m X 1m with a buffer of 0.5m in between. Water from the plots was drained off the field one day prior to spraying and the pesticides were applied using knapsack sprayer of 9 litre capacity. Soil samples were collected using box cores measuring 5cm X 5cm X 5cm. One core of soil sample (approx 625 cubic cm) was sampled randomly from sub plots and pooled to form a composite sample. Soil samples were collected at intervals of 2HAS, 24 HAS, 48 HAS, 72 HAS, 4 DAS, 5 DAS, 7 DAS, 10 DAS, 15 DAS and 30 DAS after application. Flooding was done after 24 h and again dewatered after 72 h. Water samples were collected 48 HAS followed by 3, 5, 7, 10, 15 and 30 DAS. During the flowering stage the plant samples were also collected for estimation of residue at 2HAS, 1, 2, 3, 5, 7 and 10 DAS. The materials used and methodology followed were as in the case of paddy straw (3.2.4).

3.5.2 Field situation- II

The plant protection practices followed by rice farmers in Kuttanadu in padasekharam II.

Variety : Uma

No.	Pesticide	Stage of application	Dose
1	2,4-D	Seedling stage -17 DAS	1 kg ai ha^{-1}
2	Monocrotophos	Early tillering 30DAS	0.25 kg ai ha^{-1}
3	Triazophos	Late tillering 50DAS	0.47 kg ai ha^{-1}
4	Methyl parathion	Flowering stage 90 DAS	0.25 kg ai ha^{-1}

3.5.2.1 Dissipation studies of pesticides

Residues in soil, water and plant samples were estimated as per the protocol described in 3.5.1.1

3.5 Experiment to quantify the dermal deposit of pesticides on spray men during pesticide application

An experiment was conducted to assess the level of dermal exposure to the spray men in the rice field in Kuttanadu. The dermal exposure was assessed using pad exposure technique. Three spray men were fitted with specially made cotton pads which were prepared by packing surgical gauze compressed with two pieces of heavy filter paper designed for preparative chromatography and attached it by knitting. The pads were attached to the neck, the upper arm and fore arm. Surgical gloves were used to assess the hand exposure. The spray men were allowed to do the spraying operation for one hour.

Immediately after spraying operation, the pads were removed and placed to a wide mouthed bottle containing 100 ml of dichloromethane. The contents were shaken for 30 minutes and decanted. Again 250 ml of DCM-hexane mixture was added and kept for 24 hours. This was extracted twice with DCM-hexane mixture. The pooled solvent extracts were dried by filtering through the funnel containing anhydrous sodium sulphate. The extracts were concentrated using rotary vacuum flash evaporator till complete dryness. Then the residue was dissolved in n-hexane for estimation in GC. The hand gloves were extracted using methanol and concentrated using the rotary vacuum flash evaporator and injected to GC. Residue per unit area was calculated following the formula,

$$\text{Residue per unit area} = \frac{\text{Sample peak area}}{\text{Standard peak area}} \times \text{Wt of } \frac{\text{Std} \times \text{Vol of extract}}{\mu\text{l injected} \times A_p}$$

A_p - the area of exposure of pads

The dermal exposure as residue per unit area per hour (mg man^{-2} h^{-1}) was determined by dividing the value by the time of exposure. The dermal exposure (mg man^{-1} h^{-1}) for the three spray men were determined by multiplying the value by the weight of the spray man. The dermal exposure (mg man^{-1} day^{-1}) for the three spray men were determined by multiplying the value by the total working hours of the day involved in spraying operation.

RESULTS

4. RESULTS

The salient results of the study “Impact of pesticides on abiotic and biotic environment of rice ecosystem in Kuttanadu is presented below under the following heads.

4.1 SURVEY

Survey on pesticide usage pattern and pesticide consumption in Kuttand were carried out in the rice growing areas of Kuttanadu as described in para 3.1.

3.1.2 Pesticide use pattern in Kuttanadu

Pesticide use pattern in different catchments of Kuttanadu were studied in detail during the survey. The data presented in Table 1 show the commonly used pesticides in Kuttanadu. The survey revealed that 23 pesticides were popular in Kuttanadu among which 18.18 per cent are herbicides, another 18.18 per cent are fungicides and 65.22 per cent are insecticides.

Among the different pesticides, 8.70 per cent belonged to the group ‘less toxic’, 39.13 per cent to the group ‘moderately toxic’, another 39.13 per cent to the group ‘highly toxic’and 17.39 per cent to ‘extremely toxic’.

Among the herbicides 2, 4-D was the most commonly used herbicide by the farmers in Kuttanadu. Among the farmers, 88.83 per cent followed the practice of applying 2, 4-D at 17- 20DAS at the rate

Table 1. Details of pesticides used by rice farmers in Kuttanadu rice ecosystem

No	Stage of crop	Pesticides	Rate of application (kg ai ha^{-1})	Category	Hazard category	Colour code	% of farmers using
1	Before land preparation	Glyphosate	2.00	Herbicide	Moderately toxic	Blue	23.33
2	10-12 DAS	Cyhalofop butyl	0.075-0.80	Herbicide	Moderately toxic	Blue	30.83
3	17 - 20 DAS	2,4-D sodium salt	0.8-1.00	Herbicide	Highly toxic	Yellow	88.33
4	17 - 20 DAS	Metsulfuron methyl and Chlorimuron ethyl	0.008	Herbicide	Moderately toxic	Blue	15.00
5	Seedling, Tillering	Quinalphos	0.25-0.5	Insecticide	Highly toxic	Yellow	43.33
6	Seedling, Tillering	Dimethoate	0.18	Insecticide	Highly toxic	Yellow	27.50
7	Seedling, Tillering	Imidacloprid	0.02-0.25	Insecticide	Highly toxic	Yellow	15.00
8	Seedling, Tillering	Monocrotophos	0.25-0.50	Insecticide	Extremely toxic	Red	57.50
9	Tillering	Phorate	1.00	Insecticide	Extremely toxic	Red	26.67
10	Tillering	Carbofuran	0.750	Insecticide	Extremely toxic	Red	17.50
11	Tillering and Need based	Flubendamide	0.025	Insecticide	Less toxic	Green	15.00

12	Tillering and Need based	Chlorpyriphos	0.25	Insecticide	Highly toxic	Yellow	45.83
13	Tillering and Need based	Cartap hydrochloride	0.75-1.00	Insecticide	Highly toxic	Yellow	56.67
14	Tillering and Need based	Acephate	0.500-0.750	Insecticide	Moderately toxic	Blue	73.33
15	Tillering and Need based	Triazophos	0.250-0.500	Insecticide	Highly toxic	Yellow	18.33
16	Tillering, Flowering	Lambda cyhalothrin	0.0125	Insecticide	Highly toxic	Yellow	62.50
17	Tillering, Flowering	Carbaryl	2.00	Insecticide	Highly toxic	Yellow	21.67
18	Tillering, Flowering	Malathion	2.00	Insecticide	Moderately toxic	Blue	15.00
19	Flowering	Methyl parathion	0.50	Insecticide	Extremely toxic	Red	24.17
20	Tillering	Carbendazim	0.50	Fungicide	Less toxic	Green	45.83
21	Tillering	Hexaconazole	0.05	Fungicide	Moderately toxic	Blue	68.33
22	Tillering, Flowering	Mancozeb	2.00	Fungicide	Moderately toxic	Blue	37.50
23	Ediphenphos	Hinosan	2.00	Fungicide	Moderately toxic	Blue	15.83

of 0.8-1.0 kg ai ha^{-1} . Other herbicides being used were cyhalofop butyl (30.83%) and almix (15 %). Glyphosate was applied by 23.33 per cent of farmers before field preparation.

Among the insecticides acephate (73.33%) was the most popular insecticide in the three different catchments followed by lambdacyhalothrin (62.50 %), monocrotophos (57.50 %) and cartap hydrochloride (56.67 %). Other popular insecticides were chlorpyriphos (45.83 %), quinalphos (43.33 %), dimethoate (27.50 %), phorate (26.67 %), methyl parathion (24.17 %) and carbaryl (21.67 %).

Fungicides are usually applied need based in majority of area under study. Hexaconazole (68.33 %) was reported as the most popular fungicide followed by carbendazim (45.83 %), mancozeb (37.5 %) and ediphenphos (15.83 %). In catchment of Pampa and Manimala application of hexaconazole is a common practice. Hexaconazole at the rate 0.05 kg ai ha^{-1} is applied along with acephate (0.5 kg ai ha^{-1}) at maximum tillering stage. This practice was followed even though there were no symptoms of pest or disease attack.

The pesticide use pattern in different catchments is represented in Figures 1 to 3.

The pesticide use pattern in a padashekharam with high pesticide use profile in catchment of Pampa is presented in Figure 1. The herbicide 2, 4-D was applied 17-20 DAS, followed by 2-3 applications

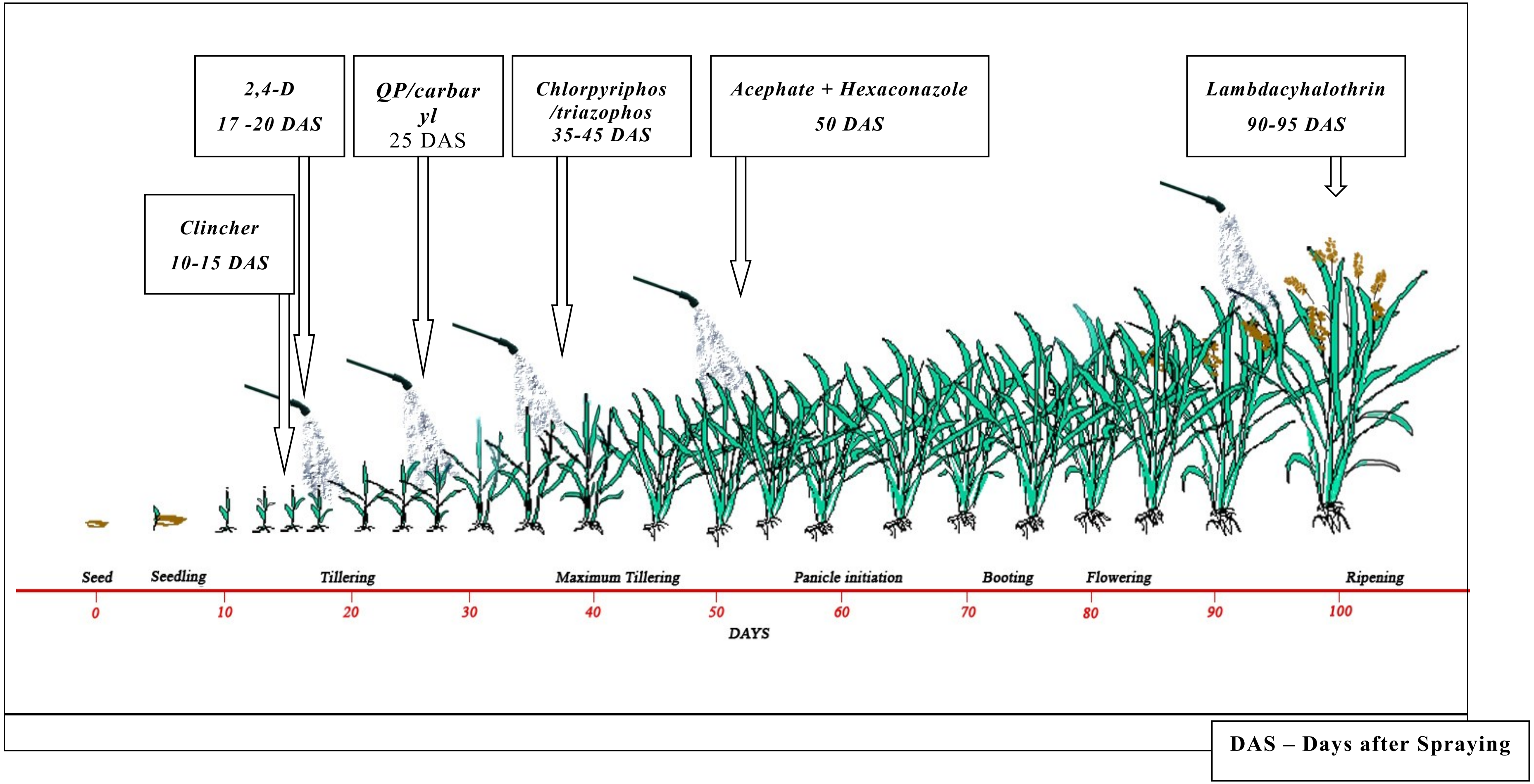


Figure 1. Pesticide use pattern in catchment of Manimala, Location: Ramankari

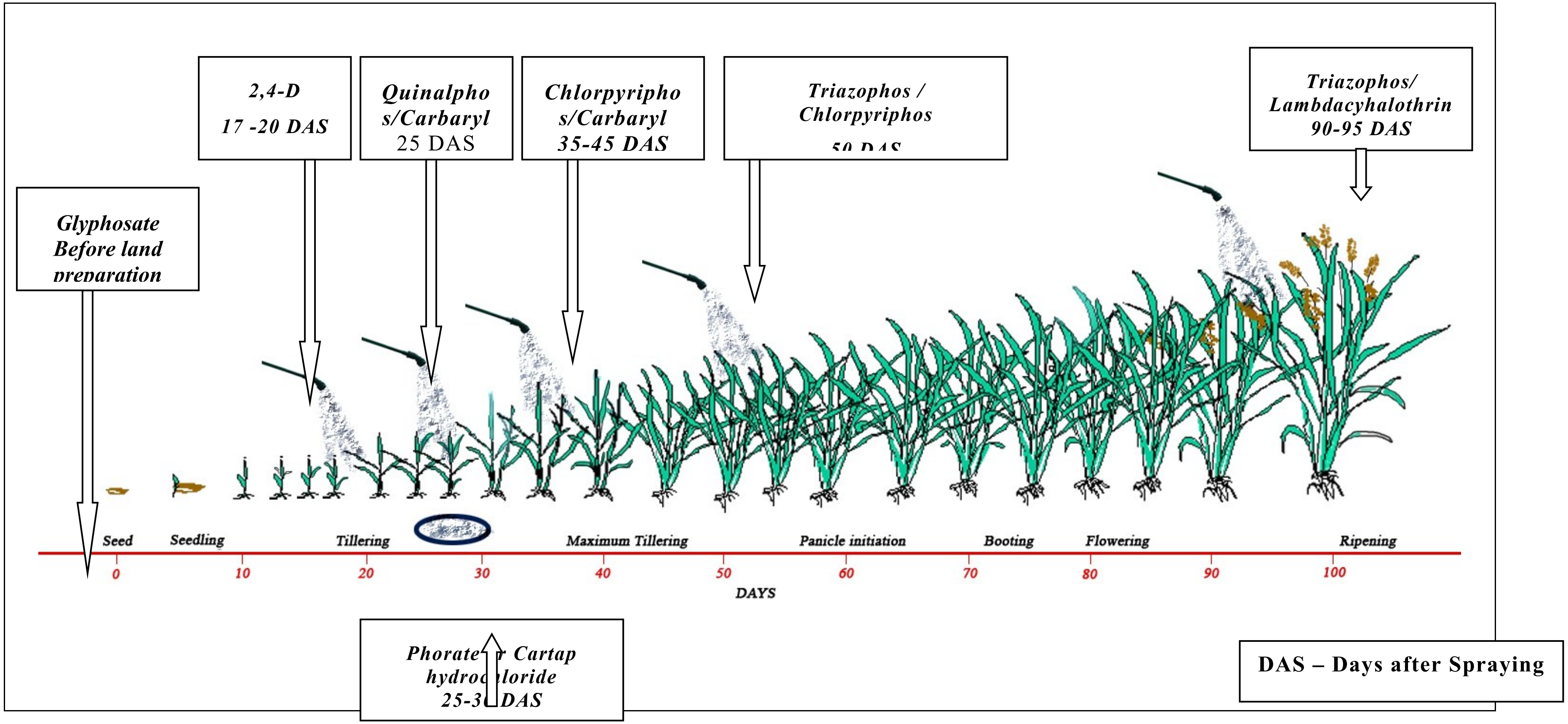


Figure 2. Pesticide use pattern in catchment of Meenachil, Location: Kumarakom

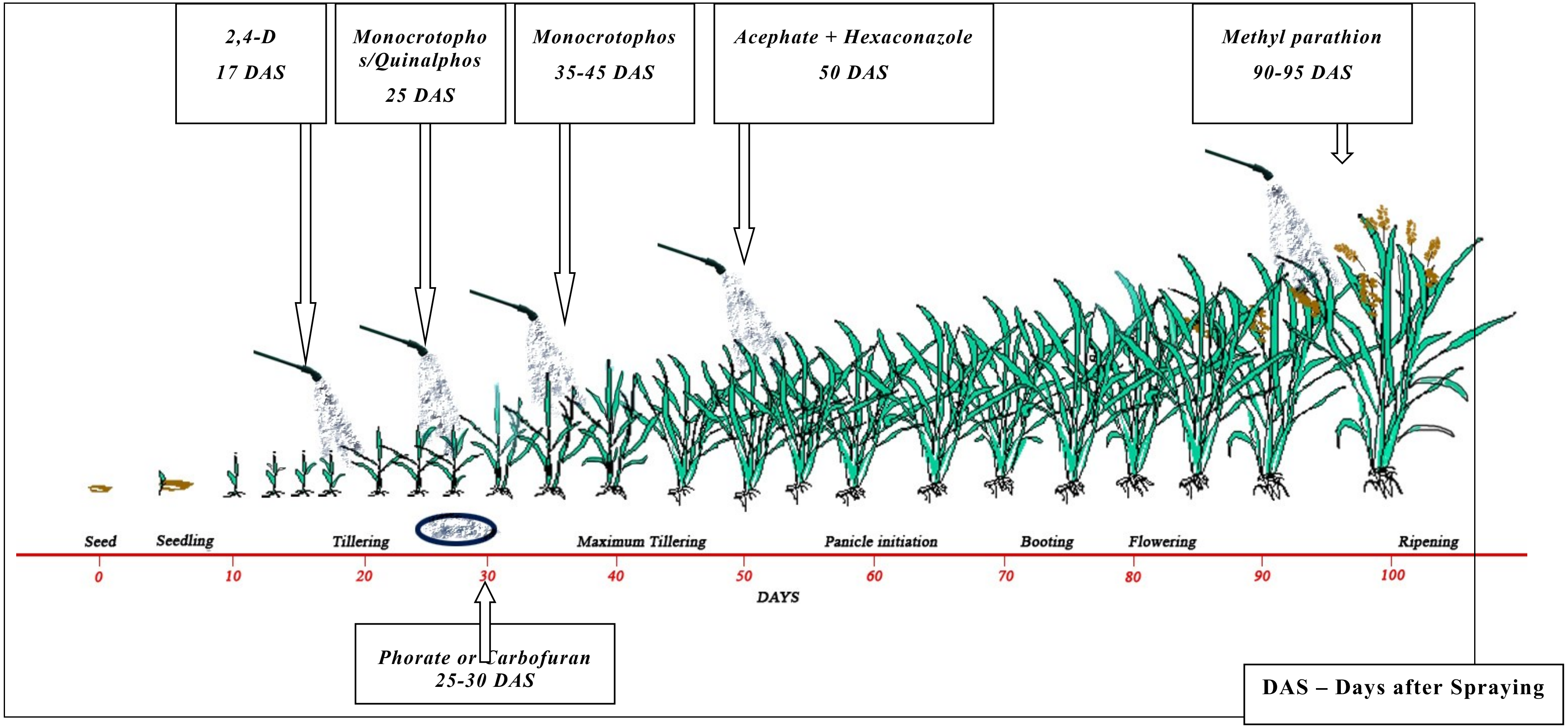


Figure 3. Pesticide use pattern in catchment of Pampa, Location : Edathua

of monocrotophos during the tillering stage. The practice of applying cartap hydrochloride or phorate at 25-30DAS was a common practice. At 50 DAS combination treatment of acephate and hexaconazole was commonly practiced. Methyl parathion was applied during the flowering stage to prevent rice bug infestation. Figure 2 represents the pesticide use pattern in a padashekharam with high pesticide use profile in catchments of Manimala. The herbicides cyhalofop butyl and 2, 4-D were applied at 10-15 DAS and 17-20 DAS. Cartap hydrochloride was applied at 25-30 DAS. Contact insecticides like quinalphos or carbaryl or chlorpyriphos were applied during the tillering stage. During the maximum tillering stage 50 DAS, combination treatment of acephate and hexaconazole was commonly practiced. During the flowering stage lambda cyhalothrin was commonly applied.

The pesticide use pattern in a padashekharam with high pesticide use profile in catchment of Meenachil is presented in Figure 3. The herbicides glyphosate was applied before field preparation. Application of 2, 4-D was practised at 17-20 DAS. Phorate or Cartap hydrochloride was applied at 25-30 DAS. Contact insecticides like chlorpyriphos and systemic insecticides like triazophos were applied during the tillering stage. During the flowering stage lambda cyhalothrin or triazophos was commonly applied.

3.1.3 Data on Pesticide consumption

The pesticide consumption in padashekarams of different catchments was found and expressed in terms of kg ai ha $^{-1}$. The frequencies of pesticide application for two consequent years 2007-08, 2008-09 and for 2002-03 were recorded. Location wise mean frequency of pesticide application was also worked out.

4.1.2.1. Quantity of pesticide application

The mean consumption of herbicides, insecticides and fungicides of the year 2007-08 is presented in Table 2. The per hectare consumption of total pesticides in different padashekarams in the catchments under study ranged from 0.560 to 3.38 kg ai ha^{-1}. The mean consumption of pesticides was highest in padashekaram in Kidangara (3.38 kg ai ha^{-1}) closely followed by Kumarakom (3.37 kg ai ha^{-1}).

In catchment of Pampa, the mean pesticide consumption was highest in Neerattupuram (2.82 kg ai ha^{-1}) followed by Edathua (2.34 kg ai ha^{-1}), Thakazhy (1.63 kg ai ha^{-1}) and Karumadi (0.56 kg ai ha^{-1}). In catchment of Manimala the padasekharam in Kidangara (3.38 kg ai ha^{-1}), recorded the highest consumption of pesticides followed by Nedumudi (2.78 kg ai ha^{-1}),Ramankari (2.643 kg ai ha^{-1}) and Pallathuruthy(2.55 kg ai ha^{-1}). In catchment of Meenachil padashekram at Kumarakom (3.36 kg ai ha^{-1}) recorded the highest pesticide consumption followed by Thiruvarpu (2.90 kg ai ha^{-1}), Vechoor (2.21 kg ai ha^{-1}), and Vaikom (1.33 kg ai ha^{-1}).

Table 2. Pesticide consumption (kg ai ha^{-1}) in padashekarams in three catchments of Kuttanadu (2007-08)

Padasekarams	Herbicide kg ai ha^{-1}	Insecticide kg ai ha^{-1}	Fungicide kg ai ha^{-1}	Total kg ai ha^{-1}
Catchment of Pampa				
Neerattupuram	1.102 (38.81)	0.750 (26.63)	0.973 (25.67)	2.817
Edathua	1.007 (42.96)	0.667 (28.46)	0.670 (22.24)	2.343
Thakazhy	0.833 (51.23)	0.753 (46.31)	0.040 (2.40)	1.627
Karumadi	0.353 (63.04)	0.090 (16.07)	0.117 (17.28)	0.56
Catchment of Manimala				
Kidangara	1.600 (47.38)	0.810 (23.99)	0.967 (22.26)	3.377
Ramankari	1.267 (47.92)	0.580 (21.94)	0.797 (23.17)	2.643
Nedumudy	1.173 (42.15)	0.943 (33.88)	0.667 (19.33)	2.783
Pallathuruthy	1.233 (48.35)	0.800 (31.37)	0.517 (16.86)	2.55
Catchment of Meenachil				
Thiruvarpu	1.633 (56.37)	0.947 (32.69)	0.317 (9.86)	2.897
Kumarakom	1.600 (47.62)	0.983 (29.26)	0.777 (18.78)	3.36
Vechoor	0.780 (35.29)	0.880 (39.82)	0.550 (19.93)	2.21
Vaikom	0.860 (49.06)	0.600 (34.23)	0.293 (18.02)	1.333

Figures in parenthesis are percentage values

The mean per hectare consumption of herbicide in different padashekarams in the catchments under study ranged from 0.353 to 1.63 kg ai ha^{-1}. The mean consumption of herbicides was highest in padashekaram in Neerattupuram and lowest in padashekaram in Karumadi. In catchment of Pampa, the mean herbicide consumption was highest in Neerattupuram (1.10 kg ai ha^{-1}) followed by Edathua (1.01 kg ai ha^{-1}), Thakazhy (0.83 kg ai ha^{-1}) and Karumadi (0.35 kg ai ha^{-1}). In catchment of Manimala the padasekharam in Kidangara (1.60 kg ai ha^{-1}), recorded the highest consumption of herbicides followed by Ramankari (1.27 kg ai ha^{-1}), Nedumudi (1.17 kg ai ha^{-1}) and Pallathuruthy(1.23 kg ai ha^{-1}). In catchment of Meenachil padashekram at Thiruvarpu (1.63 kg ai ha^{-1}) recorded the highest herbicide consumption followed by Kumarakom (1.60kg ai ha^{-1}), Vechoor (0.78 kg ai ha^{-1}), and Vaikom (0.86 kg ai ha^{-1}).

The mean per hectare consumption of insecticides in different padashekarams in the catchments under study ranged from 0.090 to 0.98 kg ai ha^{-1}. The mean consumption of insecticides was highest in padashekaram in Kumarakom (0.98 kg ai ha^{-1}) and lowest in padashekaram in Karumadi (0.09 kg ai ha^{-1}). In catchment of Pampa, the mean insecticides consumption was highest in Neerattupuram (0.75 kg ai ha^{-1}) followed by Edathua (0.67 kg ai ha^{-1}), Thakazhy (0.75 kg ai ha^{-1}) and Karumadi (0.090 kg ai ha^{-1}). In catchment of Manimala, the padasekharam in Nedumudi (0.94 kg ai ha^{-1}), recorded the highest consumption of insecticides followed by Kidangara (0.81 kg ai ha^{-1}), Pallathuruthy (0.80 kg ai ha^{-1}) and Ramankari (0.58kg ai ha^{-1}). In catchment of Meenachil padashekram at Kumarakom (0.98 kg ai ha^{-1}) recorded the highest insecticide consumption followed by Thiruvarpu (0.95kg ai ha^{-1}), Vechoor (0.88 kg ai ha^{-1}), and Vaikom (0.60 kg ai ha^{1}). The mean per hectare consumption of fungicides in different padashekarams in the catchments under study ranged from 0.04 to 0.97 kg ai $ha^{-1.}$ The mean consumption of fungicides was highest in padashekaram in Neerattupuram and Kidangara and lowest in padashekaram in Thakazhy. In catchment of Pampa, the mean fungicides consumption was highest in Neerattupuram (0.97 kg ai per ha) followed by Edathua (0.67 kg ai ha^{-1}), Karumadi (0.12 kg ai ha^{-1}) and Thakazhy (0.04 kg ai ha^{-1}). In catchment of Manimala the padasekharam in Kidangara (0.97 kg ai ha^{-1}), recorded the highest consumption of fungicides followed by Ramankari (0.80 kg ai ha^{-1}), Nedumudi (0.67 kg ai ha^{-1}) and Pallathuruthy (0.517 kg ai ha^{-1}). In catchment of Meenachil, padashekharam at Kumarakom (0.78 kg ai ha^{-1}) recorded the highest fungicide consumption followed by Vechoor (0.55 kg ai ha^{-1}), Thiruvarpu (0.32 kg ai ha^{-1}), and Vaikom (0.29 kg ai ha^{-1}).

4.1.2.2. Frequency of pesticide application

The data on frequency of pesticide application (herbicide, insecticide and fungicide) during the main crop season in Kuttanadu for the year 2007-08, 2008-09 and 2002-03 are presented in Table 3. During the year 2007-08, it was observed that 61.67 per cent of the respondents adopted one time application of herbicide during the crop season under study. Among the 120 farmers surveyed 33.33 percent of the farmers applied herbicides twice while 0.83 percent applied herbicides thrice in the particular season under study. The survey revealed that 4.17 percent farmers did not resort to any type of herbicide application in rice fields. When the catchments are examined individually frequency of herbicide application was high in catchment of Meenachil where 52.5 percent of farmers adopted two times application of herbicides while in catchment of Pampa and catchment of Manimala, the corresponding percentages were 17.50 and 30.00 respectively. In Catchment of Pampa and catchment of Manimala, 75.00 percent and 70.00 per cent of farmers adopted one time application of herbicides.

Among the 120 farmers 35.83 percent adopted one time application of insecticides while 21.67 percent farmers applied insecticides twice during the crop season under study. Ten percent farmers were observed to use insecticides three times in their field and 3.33 per cent of the farmers applied more than twice. No insecticide was used by 29.17 percent of farmers. Frequency of insecticide use was more in catchment of Manimala, where 35.00 percent of farmers adopted two time application and 15.00 percent adopted three times application.

Insecticides were applied even up to four times in the same season by 2.50 percent and 7.50 percent in catchment of Pampa and Manimala respectively.

Similarly, majority (68.33 %) of the farmers limited fungicide application to once in the crop season. Among the farmers surveyed 23 (19.17 %) farmers were not using any fungicide at all while 12.50 percent farmers were applying fungicides twice per crop season. Frequency of fungicide application was the lowest in Catchment of Pampa where 32.50 percent farmers totally avoided fungicides and 65.00 percent adopted single application .In catchment of Manimala and Meenachil, two times application of fungicides was adopted by 15.00 percent and 20.00 percent farmers respectively.

The data on frequency of pesticide application (herbicide, fungicide and insecticide) during the main crop season in Kuttanadu for the year 2008-09 is also presented in Table 3. It was observed that 65.83 percent of the respondents adopted one time application of herbicide during the crop season under study. Among the 120 farmers 19.17 percent of the farmers applied herbicides twice while none applied herbicides more than two times in the particular season under study. The survey revealed that 15.00 percent of farmers did not resort to any type of herbicide application in their rice fields. Among the 40 farmers in catchment of Pampa and Manimala, 62.50 percent each were adopting single application of herbicides. Two times application of

Table 3. Frequency of pesticide application in different catchments in Kuttanadu

No. of pesticide applications	2007-08				2008-09				2002-03			
	CI	CII	CIII	Total	CI	CII	CIII	Total	CI	CII	CIII	Total
Herbicide												
0	7.50	0.00	5.00	4.17	17.50	20.00	7.50	15.00	55.00	50.00	42.50	49.17
1	75.00	70.00	40.00	61.67	62.50	67.50	67.50	65.83	45.00	50.00	52.50	49.17
2	17.50	30.00	52.50	33.33	20.00	12.50	25.00	19.17	0.00	0.00	5.00	1.67
>2	0.00	0.00	2.50	0.83	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Insecticide												
0	30.00	17.50	40.00	29.17	20.00	10.00	40.00	10.00	17.50	0.00	5.00	7.50
1	42.50	25.00	40.00	35.83	42.50	37.50	40.00	40.00	27.50	15.00	55.00	32.50
2	22.50	35.00	7.50	21.67	32.50	47.50	7.50	40.00	30.00	47.50	35.00	37.50
3	2.50	15.00	12.50	10.00	2.50	0.00	12.50	3.33	17.50	27.50	5.00	16.67
>3	2.50	7.50	4.00	3.33	2.50	5.00	4.00	6.67	7.50	10.00	0.00	5.83
Fungicide												
0	32.50	10.00	15.00	19.17	32.50	27.50	12.50	24.17	35.00	12.50	10.00	19.17
1	65.00	75.00	65.00	68.33	67.50	55.00	67.50	63.33	52.50	55.00	47.50	51.67
2	2.50	15.00	20.00	12.50	0.00	17.50	20.00	12.50	12.50	32.50	42.50	29.17
>2	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

No. of respondents in each catchment :40
CI: Catchment of Pampa, CII: Catchment of Manimala ,CIII: Catchment of Meenachil

herbicides was adopted by 20.00 percent of farmers in catchment of Pampa while that in catchment of Manimala and Meenachil were 12.50 percent and 25.00 per cent respectively. Herbicides were never applied by 17.5 percent farmers in catchment of Pampa, 20.00 percent farmers in catchment of Manimala and 7.50 percent farmers in catchment of Meenachil .

During the year 2008-09, among the 120 farmers 40.00 percent adopted one time application of insecticide while another 40.00 percent farmers applied insecticides two times during the crop season under study. Ten per cent farmers were not using any insecticides in their field and 3.33 percent of the farmers applied more than two times. Insecticides were applied more than three times by 6.67 percent of farmers of farmers. Frequency of insecticide use was more in catchment of Manimala where 47.50 percent of farmers adopted two time application and 5.00 percent adopted more than three times application. Insecticides were applied even up to four times in the same season by 2.50 percent farmers in catchment of Pampa also.

While considering the fungicide application, it was observed that majority (63.33 %) of the farmer's limited fungicide application to one time in the crop season. Among the 120 farmers in the catchments, 29.00 (24.17 %) farmers were not using any fungicides at all while 15.00 (12.50 %) farmers were applying fungicides twice during one crop season. Frequency of fungicide application was lowest in catchments of Pampa where 32.50 percent farmers totally avoided fungicides and 67.50 percent adopted single application. In catchments of Pampa and Manimala, two times application of fungicides was adopted by 17.50 percent and 20.00 percent farmers respectively.

The frequency of pesticide application by the same farmers during the year 2002-03 (before five years) was also collected during the survey (Table 3). Among the 120 farmers surveyed, 49.17 percent had not been using any herbicide at all, another 49.17 percent adopted one time application of herbicide during the crop season. Only 1.67 percent of farmers applied herbicides twice during one crop season. Frequency of herbicide application was more or less same in the three catchments. In catchment of Meenachil, 52.50 percent of farmers adopted one time application of herbicides while in catchment of Pampa and Manimala the corresponding percentage were 45.00 and 50.00 respectively. In catchment of Meenachil, 5.00 percent of farmers applied herbicides thrice during the crop season.

Among the 120 farmers 32.50 percent adopted one time application of insecticide while 37.50 percent farmers applied insecticides twice during the crop season. Insecticides were not applied by 7.50 percent of farmers while 16.67 percent farmers used insecticides thrice in their field. Among the 120 farmers, 5.83 percent applied insecticides more than three times. In catchment of Pampa insecticides were applied once by 27.50 percent of farmers, twice by 30.00 percent and thrice by 17.50 per cent of farmers. In catchment of Manimala among 40 farmers, six (15 %) were using insecticide once, while 19 (47.50 %) using twice and 11 (27.50 %) using thrice during one crop season. In catchment of Manimala, the corresponding percent age was 32.5, 37.5 and 16.6 respectively. In catchment of Manimala, ten percent of farmers applied insecticides more than three times during one season.

Fungicide application was limited to once in a crop season by 51.67 percent of farmers. Among 120 farmers 35 (19.17 %) farmers were not using any fungicides at all while 29.17 percent farmers were applying fungicides twice during one crop season. Frequency of fungicide application was lowest in catchment of Pampa where 35.00 per cent farmers totally avoided fungicides and 52.50 per cent adopted one time application In catchment of Pampa 12.50 per cent farmers applied fungicides two times during one crop season. In catchment of Manimala and Meenachil, one time application of fungicide was adopted by 55.00 percent and 47.50 per cent while two times application was adopted by 32.50 per cent and 42.50 per cent farmers respectively.

The mean number of application of pesticides in different locations in the catchment of Pampa, Manimala and Meenachil during 2007-08, 2008-09 and 2002-03 was calculated from the above data and are presented in Table 4 to Table 6.

In case of catchment of Pampa(Table 4), the mean number of pesticide application was at Neerattupuram was 3.40 ± 1.075 during the

Table 4. Number of pesticide application in different locations of the catchment of Pampa in Kuttanadu

Locations	Year	Herbicide	Insecticide	Fungicide	Total
		Mean number of applications per season			
Neerattupuram	2007-08	1.30 ± 0.483	1.40 ± 0.699	0.70± 0.48	3.40 ± 1.075
	2008-09	1.50 ± 0.587	1.50 ± 0.527	0.60 ± 0.483	3.70 ± 0.675
	2002-03	0.50 ± 0.516	2.10 ± 0.737	0.60 ± 0.516	3.10 ± 1.197
Edathua	2007-08	1.20 ± 0.422	1.20 ± 0.919	0.90 ± 0.568	3.30 ± 0.949
	2008-09	1.30 ± 0.483	1.40 ± 0.699	0.80 ± 0.422	3.50 ± 0.707
	2002-03	0.80 ± 0.422	2.20 ± 1.033	1.10 ± 0.738	4.10 ± 1.197
Thakazhy	2007-08	1.20 ± 0.422	1.30 ± 1.16	0.60 ± 0.516	3.10 ± 1.370
	2008-09	0.80 ± 0.400	1.10 ± 0.740	0.70 ± 0.500	3.00 ± 1.00
	2002-03	0.50 ± 0.527	2.20 ± 1.135	1.00 ± 0.667	3.70 ± 1.567
Karumadi	2007-08	0.70 ± 0.483	0.30 ± 0.483	0.60 ± 0.516	1.60 ± 0.516
	2008-09	0.50 ± 0.527	1.00 ± 1.00	0.60 ± 0.500	1.60 ± 0.843
	2002-03	0.20 ± 0.421	0.30 ± 0.483	0.40 ± 0.516	0.90 ± 0.737

year 2007-08, 3.70 ± 0.675 during 2008-09 while it was 3.10 ± 1.197 in 2002-03. The mean number of herbicide applications were 1.30 ± 0.483, 1.50 ± 0.587 and 0.50 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 1.40 ± 0.699, 1.50 ± 0.527 and 2.10 ± 0.737 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 0.70 ± 0.480, 0.6 ± 0.483 and 0.6 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03.

At Edathua the mean number of pesticide application was 3.30 ± 0.949, 3.50 ± 0.707, 4.10 ± 1.197 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 1.20 ± 0.422, 1.30 ± 0.483 and 0.8 ± 0.422 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 1.20 ± 0.919, 1.40 ± 0.699 and 2.20 ± 1.033 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 0.90 ± 0.568, 0.80 ± 0.422 and 1.10 ± 0.738 respectively for the years 2007-08, 2008-09 and 2002-03.

At Thakazhy, the mean number of pesticide applications were 3.10 ± 1.370, 3.00 ± 1.00, 3.70 ± 1.567 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 1.20 ± 0.422, 0.80 ± 0.400 and 0.5 ± 0.527 respectively for the years 2007-08, 2008-09 and 2002-03 respectively.

The mean number of insecticide applications were 1.30 ± 1.06, 1.10 ± 0.740 and 2.20 ± 1.135 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 0.60 ± 0.516, 0.7 ± 0.500 and 1.00 ± 0.667 respectively for the years 2007-08, 2008-09 and 2002-03.

At Karumadi the mean number of pesticide applications were very low the values being 1.60 ± 0.516, 1.60 ± 0.843, 0.90 ± 0.737 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 0.7 ± 0.483, 0.50 ± 0.527 and 0.20 ± 0.421 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 0.30 ± 0.483, 1.00 ± 1.00 and 0.3 ± 0.483 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 0.60 ± 0.516, 0.6 ± 0.500 and 0.4 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03.

In case of catchment of Manimala, the mean number of pesticide application was at Kidangara was 4.400 ± 1.174 during the year 2007-08, 3.40 ± 0.699 during 2008-09 while it was 4.40 ± 1.070 in 2002-03. The mean number of herbicide applications were 1.50 ± 0.527, 1.10 ± 0.568 and 0.4 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 2.00 ± 0.943, 1.70 ± 0.675 and 2.9 ± 0.875 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide

Table 5. Number of pesticide application in different locations of the catchment of Manimala in Kuttanadu

Locations	Year	Herbicide	Insecticide	Fungicide	Total
		Mean number of applications per season			
Kidangara	2007-08	1.50 ± 0.527	2.00 ± 0.943	0.90 ± 0.316	4.40 ± 1.174
	2008-09	1.10 ± 0.568	1.70 ± 0.675	0.60 ± 0.516	3.40 ± 0.699
	2002-03	0.40 ± 0.516	2.90 ± 0.875	1.10 ± 0.737	4.40 ± 1.07
Ramankari	2007-08	1.10 ± 0.568	1.70 ± 0.675	0.60 ± 0.516	3.40 ± 0.699
	2008-09	0.90 ± 0.568	1.40 ± 0.699	0.40 ± 0.516	2.70 ± 1.059
	2002-03	0.60 ± 0.516	2.30 ± 0.949	0.80 ± 0.632	3.70 ± 1.49
Nedumudy	2007-08	1.20 ± 0.422	1.70 ± 1.250	1.20 ± 0.420	4.10 ± 1.370
	2008-09	0.60 ± 0.500	1.60 ± 0.970	1.10 ± 0.600	3.00 ± 1.000
	2002-03	0.40 ± 0.516	2.20 ± 0.632	1.40 ± 0.516	4.00 ± 1.247
Pallathuruthy	2007-08	1.20 ± 0.422	1.10 ± 1.197	1.40 ± 0.516	3.70 ± 1.337
	2008-09	1.10 ± 0.568	1.00 ± 1.00	1.50 ± 0.500	3.80 ± 1.032
	2002-03	0.60 ± 0.516	1.90 ± 0.737	1.50 ± 0.527	4.00 ± 0.942

applications were 0.90 ± 0.316, 0.6 ± 0.516 and 1.10 ± 0.737 respectively for the years 2007-08, 2008-09 and 2002-03.

At Ramankari the mean number of pesticide application was 3.40 ± 0.699, 2.70 ± 1.059, 3.70 ± 1.490 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 1.10 ± 0.568, 0.90 ± 0.568 and 0.60 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 1.20 ± 0.919, 1.40 ± 0.699 and 2.20 ± 1.033 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 1.70 ± 0.675, 1.40 ± 0.699 and 2.30 ± 0.949 respectively for the years 2007-08, 2008-09 and 2002-03.

At Nedumudy, the mean number of pesticide applications were 4.10 ± 1.370, 3.00 ± 1.00, 4.00 ± 1.247 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 1.20 ± 0.422, 0.60 ± 0.500 and 0.4 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 1.700 ± 1.250, 1.60 ± 0.970 and 2.20 ± 0.632 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 1.200 ± 0.420, 1.10 ± 0.600 and 1.40 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03.

At Pallathuruthy the mean number of pesticide applications were very low the values being 4.00 ± 1.247, 3.70 ± 1.337, 3.80 ± 1.032 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications was 1.20 ± 0.422 1.10 ± 0.568 and 0.60 ± 0.516, respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were, 1.10 ± 1.197, 1.00 ± 1.00 and 1.90 ± 0.737 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 1.40 ± 0.516, 1.50 ± 0.500 and 1.50 ± 0.527 respectively for the years 2007-08, 2008-09 and 2002-03.

In case of catchment of Meenachil, the mean number of pesticide application was at Thiruvarpu was 4.400 ± 0.699 during the year 2007-08, 3.90 ± 1.101 during 2008-09 while it was 4.10 ± 1.197 in 2002-03. The mean number of herbicide applications were 1.80 ± 0.422, 1.40 ± 0.516 and 0.5 ± 0.527 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 2.10 ± 0.876, 1.90 ± 0.737 and 2.60 ± 0.843 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 0.50 ± 0.3527, 0.6 ± 0.699 and 1.00 ± 0.816 respectively for the years 2007-08, 2008-09 and 2002-03.

At Kumarakom the mean number of pesticide application was 4.90 ± 1.729, 4.70 ± 949, 4.50± 0.966 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 1.40 ± 0.966, 1.30 ± 0.483 and 0.50 ± 0.527 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 2.30 ± 1.059, 2.10 ± 0.316 and 2.50 ± 0.527 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 1.20 ± 0.632, 1.30 ± 0.483 and 1.60 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03.

At Vechoor, the mean number of pesticide applications were 4.30 ± 0.949, 4.00 ± 1.00, 4.5 ± 1.354 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of herbicide applications were 1.30 ± 0.483, 1.00 ± 0.700 and 1.00 ± 0.667 respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 1.700 ± 0.989, 1.40 ± 0.700 and 2.10 ± 0.737 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 1.30 ± 0.483, 1.20 ± 0.400 and 1.40 ± 0.516 respectively for the years 2007-08, 2008-09 and 2002-03.

At Vaikom the mean number of pesticide applications were 4.40 ± 1.3547, 3.80 ± 0.789, 4.20 ± 1.317 during the years 2007-08, 2008-09 and 2002-03 respectively. The mean numbers of herbicide applications were 1.60 ± 0.516, 1.10 ± 0.568, and 0.5 ± 0.527, respectively for the years 2007-08, 2008-09 and 2002-03 respectively. The mean number of insecticide applications were 1.60 ± 1.080, 2.00 ± 1.00 and 2.40 ±

Table 6. Number of pesticide application in different locations of the catchment of Meenachil in Kuttanadu

Locations	Year	Herbicide	Insecticide	Fungicide	Total
		Mean number of applications per season			
Thiruvarpu	2007-08	1.80 ± 0.422	2.10 ± 0.876	0.50 ± 0.527	4.40 ± 0.699
	2008-09	1.40 ± 0.516	1.90 ± 0.737	0.60 ± 0.699	3.90 ± 1.101
	2002-03	0.50 ± 0.527	2.60 ± 0.843	1.00 ± 0.816	4.10 ± 1.197
Kumarakom	2007-08	1.40 ± 0.966	2.30 ± 1.059	1.20 ± 0.632	4.90 ± 1.729
	2008-09	1.30 ± 0.483	2.10 ± 0.316	1.30 ± 0.483	4.70 ± 0.949
	2002-03	0.50 ± 0.527	2.50 ± 0.527	1.60 ± 0.516	4.50 ± 0.966
Vechoor	2007-08	1.30 ± 0.483	1.70 ± 0.989	1.30 ± 0.483	4.30 ± 0.949
	2008-09	1.00 ± 0.700	1.40 ± 0.700	1.20 ± 0.400	4.00 ± 1.000
	2002-03	1.00 ± 0.667	2.10 ± 0.737	1.40 ± 0.516	4.50 ± 1.354
Vaikom	2007-08	1.60 ± 0.516	1.60 ± 1.080	1.20 ± 0.422	4.40 ±1.075
	2008-09	1.10 ± 0.568	2.00 ± 1.00	1.20 ± 0.400	3.80 ± 0.789
	2002-03	0.50 ± 0.527	2.40 ± 0.516	1.30 ± 0.674	4.20 ± 1.317

0.516 respectively for the years 2007-08, 2008-09 and 2002-03. The mean number of fungicide applications were 1.200 ± 0.422,1.20 ± 0.400, and 1.30 ± 0.674 respectively for the years 2007-08, 2008-09 and 2002-03.

The various sources utilized by farmers for their knowledge regarding pesticide use is presented in Table 7. It was observed that majority of farmers (45.83 %) relied upon pesticide sales men for information regarding purchase and use of pesticides. Out of the 120 farmers 31.67 per cent depend on government personnel for their knowledge regarding pesticide and 15 per cent considered their fellow farmers as their source of information. Only 4.17 per cent farmers depend on other sources like advertisements, radio, T.V etc as their source for information for selection of pesticides while 3.33 per cent of farmers depended on pesticide labels for their knowledge regarding pesticides.

When data are examined catchment wise, it was noticed that in catchment of Pampa, 52.5 per cent of farmers relied upon pesticide sales men while 20.00 per cent each considered government personnel and fellow farmers as their source of information. Only 2.5 per cent of farmers depended on pesticide labels while 5 per cent of farmers depended on other sources for their information regarding pesticides use.

Table 7. Sources of knowledge of pesticides for rice farmers

Source	Catchment of							
	Pampa		Manimala		Meenachil		Total	
	Number	Percentage	Number	Percentage	Number	Percentage	Number	Percentage
Government personnel	8.00	20.00	19.00	47.50	11.00	27.50	38.00	31.67
Pesticide labels	1.00	2.50	2.00	5.00	1.00	2.50	4.00	3.33
Fellow farmers	8.00	20.00	6.00	15.00	4.00	10.00	18.00	15.00
Pesticide salesman	21.00	52.50	12.00	30.00	22.00	55.00	55.00	45.83
Others	2.00	5.00	1.00	2.50	2.00	5.00	5.00	4.17

In catchment of Manimala , among the 40 farmers surveyed 47.5 per cent depended on government personnel for their knowledge regarding pesticides. The pesticide sales men operated as the source of information for 30 per cent of farmers. During the survey it was revealed that 15 per cent of farmers depended on fellow farmers while only 5.00 per cent depended on pesticide labels. The remaining 2.50 per cent depended on other sources.

In catchment of Meenachil, among the 40 farmers surveyed 55 per cent depended the pesticide sales men on for their knowledge regarding pesticides. Government personnel were the source of information for 27.5 per cent of farmers. During the survey it was revealed that 10 per cent of farmers depended on fellow farmers while only 2.5 per cent depended on pesticide labels. The remaining 5.00 per cent depended on other sources.

The factors that farmers consider the most to initiate the application of insecticides were also collected and recorded (Table 8). Majority of farmers (54.17 %) applied pesticides when they observed presence of pest in the field. The date of sowing or transplanting was considered by 15.83 per cent of farmers. Farmers also consider other factors like weather conditions, application by fellow farmers etc as a factor to depend on in order to initiate spraying operations. Among the 120 farmers 19.17 per cent depend on other factors to initiate the spraying operations. The degree of pest infestation was taken into

Table 8. Criteria for commencement of pesticide application

	Catchment of							
	Pampa		Manimala		Meenachil		Total	
	Number	Percentage	Number	Percentage	Number	Percentage	Number	Percentage
Presence of pest	23.00	57.50	22.00	55.00	20.00	50.00	65.00	54.17
Degree of pest infestation	5.00	12.50	2.00	5.00	6.00	15.00	13.00	10.83
Date of Transplanting/ sowing/fertilizer application	8.00	20.00	8.00	20.00	3.00	7.50	19.00	15.83
Others	4.00	10.00	8.00	20.00	11.00	27.50	23.00	19.17

account by only 10.83 per cent of the farmers initiate pesticide application.

In catchment of Pampa 57.50 per cent of farmers resorted to spraying operations on observing the presence of pest in the field while degree of infestation is considered by only 12.5 per cent of farmers. Only 20.00 per cent of farmers depended on time of transplanting while another 10 per cent depended on other factors like weather conditions, application by fellow farmers etc for initiating spraying operations.

In catchment of Manimala, 55.00 per cent of farmers initiated spraying operations on observing the presence of pest in the field while degree of infestation is considered by only 5.00 per cent of farmers. When 20.00 per cent of farmers depended on time of transplanting, another 20 per cent depended on other factors for initiating spraying operations.

In catchment of Meenachil, 50.00 per cent of farmers initiated spraying operations on observing the presence of pest in the field. Degree of infestation is considered by 15.00 per cent of farmers. When 7.50 per cent of farmers depended on time of transplanting, another 27.5 per cent depended on other factors mentioned above.

3.1.4 Extent of adoption of GAP and IPM

Table 9 represents the extent of awareness about IPM and GAP and its adoption in farmers fields in Kuttanadu. Among the 120 farmers

Table 9. Extent of adoption of IPM and GAP in Agriculture in Kuttanadu

Component	Catchment of						Total	
	Pampa		Manimala		Meenachil			
	Aware	Unaware	Aware	Unaware	Aware	Unaware	Aware	Unaware
IPM	70.00	30.00	80.00	20.00	75.00	25.00	75.00	25.00
GAP in pesticide use	2.50	97.50	0.00	100.0	7.50	92.50	3.33	96.67
Adoption of IPM								
	Adopted	Not adopted	Adopted	Not adopted	Adopted	Not adopted	Adopted	Not adopted
IPM Adoption	45.00	55.00	37.50	62.50	35.00	65.00	39.16	60.8

No of respondents in each catchments =20 surveyed, 75.00 per cent were aware of the concept of IPM and practices to be followed. Among them only 47.00 per cent were adopting IPM techniques in their rice fields. Only 4 farmers in Kuttanadu were aware of the concept of Good Agricultural practices.

Out of the forty farmers surveyed in catchment of Pampa, seventy per cent were aware of IPM practices. Among them, only 45.00 per cent farmers were adopting IPM in their fields .Only 2.50 per cent of the farmers were aware of the concept of GAP in rice cultivation. In catchment of Manimala, 80.00 per cent were aware of IPM but only 15.00 per cent of farmers adopted IPM measures for pest management. None of them were aware of the concept of GAP in rice cultivation. In catchment of Meenachil, 75.00 per cent of the farmers were aware of IPM concept while the extent of adoption of IPM was only 35.00 per cent. Among the farmers only 7.50 per cent were aware of the concept of GAP.

4.1.4 Extent of adoption of protective measures while pesticide handling

Table 10 presents the extent of adoption of protective measures by pesticide spray men in Kuttanadu. The survey among the pesticide spraymen revealed that during pesticide handling, not a single farmer in Kuttanadu was adopting protective gadgets as per the FAO guidelines. Data collected from 60 spray men revealed that 30.00 per cent of them were not using any protective measures during pesticide

Table 10. Extent of adoption of protective measures while handling pesticides

	Catchment						Total n=60	
	Pampa		Manimala		Meenachil			
	Adopted	Not adopted	Adopted	Not adopted	Adopted	Not adopted	Adopted	Not adopted
Protective measure as per FAO guide lines	NIL	100	NIL	100	NIL	100	NIL	100
Protective measure Partially adopted	75.00	25.00	75.00	25.00	60.00	40.00	70.00	30.00
Kinds of protective cover								
Gloves	0.00	100	0.00	100	0.00	100	0.00	100.00
Boots/ footwear	5.00	95.00	0.00	100	5.00	95.00	3.33	96.67
Cover the nose	70.00	30.00	75.00	25.00	55.00	45.00	66.67	33.33

No of respondents in each catchment: 5

application while 70.00 per cent were adopting partial protective measures which include use of boots (66.67 %) and covering the nose (3.33 %.)

In catchment of Pampa, 25 per cent of the pesticide spraymen were not adopting any type of protective measures. Among them, 70.00 per cent followed the practice of covering the nose during pesticide application while 5.00 per cent were using boots.

In catchment of Manimala, also, 25 per cent of the pesticide spraymen were not adopting any type of protective measures. Among them, 75.00 per cent were covering the nose during pesticide application but none of them were using boots.

In catchment of Meenachil , 40.00 per cent of the pesticide spray men were not adopting any type of protective measures. Among them, 55.00 per cent followed the practice of covering the nose during pesticide application while 5.00 per cent were using boots.

4.1.4 Extent of adoption of safe practices in pesticide storage and disposal.

The pesticide storage and disposal practices followed by farmers in Kuttanadu are given in Table 11. The data revealed that among the 120 farmers 55.83 per cent were not following safe storage practices while 41.17 per cent were following safe storage practices. In catchment of Pampa, 55 per cent were not following safe storage

Table 11. Extent of adoption of safe practices during pesticide storage and disposal

Practices followed	Catchment of							
	Pampa		Manimala		Meenachil		Total	
	No. of respondents	Percentage	No. of respondents	Percentage	No. of respondents	Percentage	No. of respondents	Percentage
Storage practices								
Safe storage practices	18.00	45.00	15.00	37.50	20.00	50.00	53.00	41.17
Unsafe storage practices	22.00	55.00	25.00	62.50	20.00	50.00	67.00	55.83
.Disposal of empty bottles								
Bury in soil after use	2.00	5.00	3.00	7.50	2.00	5.00	7.00	5.80
Dispose in paddy ecosystem	38.00	95.00	37.00	92.50	38.00	95.00	113.00	94.16
Disposal of wash water after spraying								
In the irrigation canal	18.00	45.00	16.00	40.00	15.00	37.50	49.00	40.8
In paddy field	20.00	50.00	24.00	60.00	24.00	60.00	68.00	56.67
others	2.00	5.00	0.00	0.00	2.00	5.00	4.00	3.33

practices while in catchment of Manimala and Meenachil the corresponding values were 62.50 and 50.00 per cent respectively.

The used pesticide bottles were disposed in paddy eco system itself by 94.16 per cent of farmers. Only 5.80 per cent were burying the bottles in soil after use.In catchment of Pampa, 95.00 per cent followed the unscientifice practice while in catchment of Manimala and Meenachil the percentage values were 92.50 and 95 .00 respectively.

Among the farmers surveyed 56.67 per cent disposed the pesticide wash water in the paddy field while 40.80 per cent in the irrigation channel adjacent to paddy field. Only 3.33 per cent of farmers were disposing wash water away from paddy eco-system. In catchment of Pampa the wash water was disposed in paddy field by 50.00 per cent of farmers and in irrigation channel by 45.00 per cent of the farmers. Only 5.00 per cent of farmers disposed wash water in places other than the paddy eco-system. In catchment of Manimala, the wash water was disposed in paddy field by 60.00 per cent of farmers and in irrigation channel by 40.00 per cent of the farmers.

In catchment of Meenachil , the wash water was disposed in paddy field by 60.00 per cent of farmers and in irrigation channel by 37.50 per cent of the farmers. Only 5.00 per cent of farmers were disposing wash water in places other than paddy eco-system.

Table 12 presents the knowledge regarding label information on pesticide containers. Among the 60 spray men, 83.33 per cent opined

Table 12. Knowledge regarding label information on pesticide containers

Particulars	Percentage of respondents			
	Pampa*	Manimala*	Meenachil*	Total
Follow label information	5.00	30.00	15.00	16.67
Do not follow label information	95.00	70.00	85.00	83.33
Reasons for not following label information				
Cannot read , Too small letters	40.00	40.00	50.00	43.33
Ignorant about label information	60.00	60.00	50.00	56.67

*- Number of respondents in each area was 20.

that they were not following the label information on pesticide containers while 16.67 per cent followed the instruction given on pesticide labels. In catchment of Pampa 95.00 per cent of pesticide spray men were not following label information while only 5.00 per cent were following it. In catchment of Manimala, 70.00 per cent were not following label information while 30.00 per cent were following the label instructions. In catchment of Meenachil, 85.00 per cent were not following label information while 15.00 per cent were following it.

Among the spray men, 43.33 per cent opined that the labels were illegible and they were unable to read it while 56.67 per cent were ignorant about the pesticide labels. In catchment of Pampa 40.00 per cent of pesticide spray men opined that the labels could not be read, while only 60.00 per cent were ignorant about label information. In catchment of Manimala, 40.00 per cent of pesticide spray men opined that the labels could not be read, while only 60.00 per cent were ignorant about label information. In catchment of Meenachil, 50.00 per cent of pesticide spray men opined that the labels could not be read, while another 60.00 per cent were ignorant about label information.

4.1.5 Direct health impacts to spray men on repeated pesticide exposure

The direct health impacts to spray men on repeated pesticide exposure was collected during the survey (Table 13).Out of the sixty spray men surveyed, headache and dizziness were the main problems

Table 13. Health problems as reported by spray men in different catchments

Health problems noticed	Pampa		Manimala		Meenachil		Total	
	Number of respondents	Percent age	Number of respondent s	Percentag e	Number of respondent s	Percent age	Number of respondent s	Perce ntage
Headache, dizziness	6.00	30.00	8.00	40.00	12.00	60.00	26.00	43.33
Vomiting	3.00	15.00	1.00	5.00	0.00	0.00	4.00	6.60
Unconsciousnes s	1.00	5.00	0.00	0.00	1.00	5.00	2.00	3.33
Stomach pain	2.00	10.00	3.00	15.00	1.00	5.00	6.00	10.00
Weakness	2.00	10.00	0.00	0.00	3.00	15.00	5.00	8.30
Skin irritation	6.00	30.00	8.00	40.00	3.00	15.00	17.00	28.33

for majority (43.33 %) of the respondents. Dermal diseases on exposure to pesticides were another important health hazard. Among the sixty farmers 28.33 per cent were suffering from dermal diseases. Another health problems faced by the pesticide spray men was stomach pain (10.00 %), general weakness (8.30 %) and vomiting (6.60%). During long periods of pesticide application unconsciousness occurred in case of 3.33 per cent of spray men.

In catchment of Pampa, 30.00 per cent of the spraymen were suffering from dermal diseases. Another 30.00 per cent were suffering from headache and dizziness. Among the twenty spraymen 15.00 per cent each were suffering from vomiting while 10.00 per cent each were suffering from stomach pain and dermal diseases. Unconsciousness occurred in case of 5.00 per cent of spraymen on long exposure to pesticides.

In catchment of Manimala, 40.00 per cent of the spraymen were suffering from dermal diseases. Another 40.00 per cent were suffering from headache and dizziness. Among the twenty spraymen 15.00 per cent each were suffering stomach pain while vomiting occurred in 5.00 per cent of the spraymen. Cases of unconsciousness were not reported.

In catchment of Meenachil, 60.00 per cent of the spraymen were suffering from headache and dizziness. Among the twenty spray men 15.00 per cent each were suffering stomach pain while general weakness occurred in another 15.00 per cent of spray men. Occurrence of vomiting and unconsciousness were reported by 5.00 per cent each of the farmers in catchment of Meenachil.

4.2 **Validation of Multi Residue Methods (MRM) for pesticide residue analysis of water, soil, rice grains and paddy straw, fish and duck meat**

4.2.1 Validation of MRM for pesticide residue analysis of water

Results of the preliminary recovery studies of three different multi residue methods *viz.* Micro-separator method, modified Hernanadez method and Solid phase extraction are presented in Table 14. The mean recovery of lindane at 1 ppb level by microseparator method was 86.33 per cent while the corresponding recoveries by modified Hernanadez method and by solid phase extraction method were 92.96 and 78.32 per cent respectively. The mean recovery for methyl parathion was the highest in SPE method (91.88 %) followed by microseparator method (86.85 %) and modified Hernanadez method (80.80 %). Mean recovery per cent of chlorpyriphos was the highest (96.72) in microseparator method while the recovery in modified Hernanadez method was 84.48 and that in SPE method was 83.51.The mean recovery of alpha endosulfan was maximum (95.17 %) in modified Hernanadez method while that in microseparator method and SPE method were 94.70 and 90.80 per cent respectively. The mean per cent recovery of ethion was 94.08, 90.17 and 88.26 for Microseparator, modified Hernanadez and SPE method respectively. Results of the

Table 14. Recovery of pesticides in water following different methods
(Level of fortification :1 ppb)

Pesticides/Method	Micro separator method		Modified Hernandez Method		SPE Method	
	Recovery %	SD	Recovery %	SD	Recovery %	SD
Lindane	86.33	4.568	92.96	7.301	78.32	1.767
Methyl Parathion	86.85	5.188	80.80	3.530	91.88	0.306
Chlorpyriphos	96.72	1.998	84.48	9.180	83.51	0.764
Alpha Endosulfan	94.70	7.779	95.17	0.780	90.80	0.565
Ethion	94.08	3.863	90.17	1.160	88.26	1.113

SD- Standard Deviation

preliminary validation studies indicated that among the three methods tested, micro separator method and modified Hernanadez method gave better recovery than SPE method in most of the cases. Even though satisfactory recoveries were observed for both the methods, modified Hernanadez method was preferred for further studies as compounds like 2,4-D could not be extracted by micro separator method. Only pesticides that are soluble in n-hexane could be extracted by micro separator method. Hence modified Hernanadez method was selected for further studies on method validation.

4.2.1.1. Validation of Method of Hernandez et al. (1993) for pesticide residue analysis in water

The quality parameters for method validation of eleven pesticides prevalently used in Kuttanadu such as recovery per cent, repeatability, reproducibility, linearity, limit of detection and quantification essential to assess the method are presented in Tables 15 to 16. The pesticides in the table are given in the order of their retention time in GC which ranged from 5.74 to 35.74 minutes (Table 30). The repeatability in terms of recovery percentage of the method was determined at four levels, 100, 50, 10 and 1 ppb. The mean per cent recovery of phorate at concentrations of 100, 50, 10 and 1 ppb were 106.51, 95.18, 80.93 and 78.37 per cent respectively. The mean per cent recovery of dimethoate at levels of 100, 50 and 10 ppb were

Table 15. Repeatability of MRM of pesticides in water at four spiked levels following method of Hernandez et al.,(1993)

	Level of fortification											
	100 ppb			50 ppb			10 ppb			1ppb		
	Mean Recovery %	SD	RSDr %	Mean Recovery %	SD	RSDr %	Mean Recovery %	SD	RSDr %	Mean Recovery %	SD	RSDr %
Phorate	106.51	5.461	5.127	95.18	4.820	5.064	80.93	4.819	5.954	78.37	2.063	2.633
Dimethoate	91.47	2.029	2.218	76.13	8.657	11.371	63.82	1.83	2.87	NS	NS	NS
Lindane	96.21	3.848	3.999	96.59	9.079	9.400	98.72	4.959	5.023	95.49	10.195	10.676
Methyl parathion	103.92	6.455	6.211	99.71	12.676	12.712	107.28	5.468	5.097	80.79	3.530	4.370
Malathion	111.02	8.848	7.970	103.19	4.520	4.380	107.38	8.640	8.047	92.34	3.066	3.320
Chlorpyriphos	104.60	2.231	2.132	107.31	6.770	6.309	112.41	6.664	5.929	84.48	9.180	10.860
Alpha endosulfan	91.83	15.707	17.105	91.87	10.196	11.099	98.24	5.456	5.554	94.83	1.030	1.085
Hexaconazole	71.18	2.024	2.844	63.84	2.195	3.438	55.51	3.10	5.58	NS	NS	NS
Ediphenphos	98.47	7.482	7.598	88.52	2.336	2.639	61.26	7.280	11.885	NS	NS	NS
Lambda cyhalothrin	74.76	5.306	7.098	72.76	8.306	11.415	77.54	7.807	10.068	NS	NS	NS
Deltamethrin	70.83	6.767	9.554	69.46	1.577	2.271	72.36	7.289	10.073	NS	NS	NS

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:
RSD r - Relative standard deviation for reproducibility
NS- Not Significant

Table 16. Reproducibility of MRM of pesticides in water at three spiked levels following method of Hernandez et al.,(1993)

Pesticides	Level of fortification								
	100 ppb			50 ppb			10 ppb		
	Mean Recovery%	SD	RSD r	Mean Recovery%	SD	RSD r	Mean Recovery%	SD	RSD r
Phorate	100.50	1.500	1.493	92.47	1.120	1.211	79.98	4.525	5.658
Dimethoate	88.80	1.799	2.026	77.57	8.295	10.694	61.67	1.68	2.72
Lindane	96.99	3.605	3.717	95.66	8.936	9.342	98.42	4.930	5.010
Methyl parathion	105.06	6.141	5.845	95.75	10.656	11.129	109.24	4.284	3.922
Malathion	115.18	5.140	4.462	102.24	4.206	4.114	103.35	5.091	4.927
Chlorpyriphos	105.83	0.680	0.642	110.96	2.445	2.203	109.37	4.100	3.749
Alpha endosulfan	88.58	14.664	16.555	96.18	6.939	7.215	101.37	0.609	0.600
Hexaconazole	70.20	1.110	1.581	63.20	1.890	2.991	53.53	1.50	2.807
Ediphenphos	94.37	2.320	2.459	88.36	2.320	2.626	58.84	5.961	10.131
Lambda cyhalothrin	74.32	5.250	7.064	73.32	8.250	11.253	77.31	7.796	10.085
Deltamethrin	71.62	6.626	9.252	68.57	0.327	0.477	72.40	7.289	10.067

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:
RSD r - Relative standard deviation for reproducibility.

91.47, 76.13 and 63.82 per cent respectively. Dimethoate did not recover satisfactorily at level of 1 ppb.

At 100 ppb level of fortification, the mean per cent recovery among other pesticides were lindane (96.21), methyl parathion (103.92), malathion (111.02), chlorpyriphos (104.60), alpha endosulfan (91.83), hexaconazole (71.18), ediphenphos (98.47), lambdacyhalothrin (74.76), and deltamethrin (70.83) .

At the forticiation level of 50 ppb, the mean per cent recovery among the pesticides in the descending order were chlorpyriphos (107.31) > malathion (103.19) > methyl parathion (99.71), lindane (96.59) > phorate(95.18), > alpha endosulfan (91.87) >ediphenphos (88.52) > lambda cyhalothrin (72.76) > deltamethrin (69.46) > hexaconazole (63.84).

The mean per cent recovery values were 98.72, 107.28, 107.38, 112.41 and 98.24 for lindane, methyl parathion, malathion, chlorpyriphos and alpha endosulfan respectively at fortification level of 10 ppb. The corresponding values at 1ppb were 95.49, 80.79, 92.34, 84.48 and 94.83 for lindane, methyl parathion, malathion, chlorpyriphos and alpha endosulfan respectively.

The mean per cent recovery of hexaconazole, ediphenphos, lambdacyhalothrin and deltamethrin were 55.51, 61.26, 77.54 and 72.36 respectively at 10ppb. The repeatability of eleven pesticides were established at 100, 50 and 10 ppb level. At 1 ppb level repeatability of six pesticides alone could be established for the method under validation in the present study. The reproducibility of the method is determined by analyzing the fortified sample at three levels by following the same method over three consecutive days under same analytical conditions. The mean recovery of all the eleven pesticides under study were above 70 per cent at the higher two levels of fortification of 100 and 50 ppb. The mean recovery of the different pesticides under study ranged from 70.20 to 115.18 per cent with RSD < 20. At 10 ppb level, reproducibility could be established for all eleven pesticides with RSD < 20 %.

Results of method validation studies for estimation of 2,4-D in water are presented in Table 17. The repeatability in terms mean per cent recovery of 2,4-D at different levels of fortification were recorded as 73.35, 77.68 and 74.37 per cent at 0.1, 0.05 and 0.01 ppm respectively. The reproducibility of this herbicide at these three levels was also established. The mean per cent recovery observed on the three consecutive days of analysis were 73.42, 75.32 and 74.77 at 0.1, 0.05 and 0.01 ppm respectively.

4.2.2 Preliminary Validation of MRM for pesticide residue analysis of soil

Recovery studies were conducted in soil using two different methods viz. Soxhlet extraction method and Acetone extraction method. The results of recovery studies are presented in Table 18.

Table 17. Recovery of 2, 4-D in water following Method of Hernandez et al.,(1993)

Level of fortification (ppm)	Mean Recovery%	SD	RSDr
Repeatability			
0.1	73.35	3.069	4.184
0.05	77.68	2.897	3.729
0.01	74.37	2.122	2.853
Reproducibility			
Pesticides Concentration	Mean Recovery%	SD	RSD*r*
0.1	73.42	2.176	2.964
0.05	75.32	4.531	6.0175
0.01	74.77	1.530	2.047

Number of replicates at each level (n)= 6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days: *RSD r* - Relative standard deviation for reproducibility.

Table 18. Recovery of pesticides in soil following different methods

(Level of fortification : 0.1 ppm)

Pesticides	Soxhlet extraction method		Acetone extraction Method	
	Mean Recovery%	SD	Mean Recovery%	SD
Lindane	64.16	4.721	77.34	1.262
Methyl Parathion	65.98	5.345	88.58	1.140
Chlorpyriphos	78.06	1.510	94.74	2.009
Alpha Endosulfan	60.72	4.992	76.13	2.157
Ethion	54.96	7.075	70.79	4.547

SD-. Standard Deviation

When soxhlet extraction method was followed at level of fortification of 0.1ppm, the per cent recovery of lindane, methyl parathion, chlopyriphos, alpha endosulfan and ethion were 64.16, 65.98, 78.06, 60.72 and 54.96 respectively. In acetone extraction method, the per cent recovery was higher and the recovery per cent were 77.34, 88.58, 94.74, 76.13 and 70.79 for lindane, methyl parathion, chlopyriphos, alpha endosulfan and ethion respectively.

Since the per cent recovery was better, acetone extraction method was selected and the method was validated by establishing the validation parameters such as, repeatability, reproducibility, linearity, limit of detection and quantification.

4.2.2.1 Validation of acetone extraction method for pesticide residue analysis of soil

Data presented in Table 19 represent the mean recovery for the eleven selected pesticides at three different fortification levels of 0.2, 0.1 and 0.05 ppm. The mean recovery of chlorpyriphos, malathion, phorate, dimethoate, deltamethrin, ediphenphos, methyl parathion, alpha endosulfan, lambdacyhalothrin, lindane and hexaconazole at 0.2 ppm level were 108.87 ,104.30, 103.67, 90.15, 88.14 , 86.14, 85.06, 83.91,73.22, 73.14 and 65.18 per cent with RSD <20 % respectively.

At 0.1 ppm level, the mean per cent recovery in the descending order was chlorpyriphos (110.07), phorate (107.34), malathion (96.25), alpha endosulfan (84.78), methyl parathion (82.38),

Table 19. Repeatability of different pesticides in soil at three spiked levels (Acetone extraction Method)

Pesticide	Level of fortification								
	0.2ppm			0.1ppm			0.05ppm		
	Mean recovery%	SD	RSDr	Mean recovery%	SD	RSDr	Mean recovery %	SD	RSDr
Phorate	103.67	9.555	9.216	107.34	10.320	9.615	101.56	4.153	4.089
Dimethoate	90.15	13.520	14.997	66.77	8.763	13.125	66.05	4.586	6.943
Lindane	73.14	6.096	8.334	73.12	3.410	4.663	74.43	2.118	2.846
Methyl parathion	85.06	1.477	1.736	82.38	1.350	1.639	88.27	8.012	9.076
Malathion	104.30	7.790	7.469	96.25	13.068	13.578	89.68	5.125	5.714
Chlorpyriphos	108.87	7.536	6.922	110.07	2.604	2.366	101.96	3.938	3.863
Alpha endosulfan	83.91	5.128	6.111	84.78	3.197	3.771	81.19	3.581	4.410
Hexaconazole	65.18	3.683	5.651	62.73	2.003	3.193	NS	NS	NS
Ediphenphos	86.14	6.394	7.422	81.25	6.095	7.501	75.57	2.781	3.680
Lambda cyhalothrin	73.22	5.727	7.822	71.78	2.456	3.422	79.31	4.241	5.347
Deltamethrin	88.14	5.537	6.283	74.37	8.602	11.567	69.52	7.415	10.666

SD-. Standard Deviation
RSD - Relative standard deviation

Table 20. Reproducibility of different pesticides in soil at three spiked levels (Acetone extraction Method)

Pesticide	Level of fortification								
	0.2ppm			0.1ppm			0.05ppm		
	Mean recovery%	SD	RSD r	Mean recovery%	SD	RSD r	Mean recovery %	SD	RSD r
Phorate	100.66	2.896	2.877	103.56	6.253	6.038	104.49	6.044	5.784
Dimethoate	90.76	15.678	17.273	67.34	4.671	6.936	68.46	3.108	4.539
Lindane	76.83	6.627	8.626	73.07	1.949	2.668	76.43	1.947	2.547
Methyl parathion	84.22	1.918	2.278	82.33	0.071	0.087	87.48	8.918	10.194
Malathion	107.77	10.992	10.200	96.01	12.944	13.482	89.60	6.462	7.212
Chlorpyriphos	103.55	5.622	5.429	107.99	1.985	1.838	102.05	3.237	3.172
Alpha endosulfan	84.94	2.180	2.566	85.62	3.664	4.280	81.18	4.649	5.726
Hexaconazole	64.20	1.857	2.893	63.11	1.560	2.472	NS	NS	NS
Ediphenphos	84.27	4.272	5.069	78.78	3.291	4.177	75.69	4.212	5.564
Lambda cyhalothrin	78.48	8.320	10.601	71.50	4.245	5.938	78.69	5.568	7.076
Deltamethrin	93.46	2.469	2.641	73.63	8.135	11.047	70.45	6.983	9.912

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r Relative standard deviation for reproducibility.

ediphenphos (81.25), deltamethrin (74.37), lindane(73.12), lambda cyhalothrin (71.78), dimethoate (66.77) and hexaconazole (62.73). At 0.05 ppm, the mean recovery of the pesticides were chlorpyriphos (101.96), phorate (101.56), malathion (89.68), methyl parathion (88.27), alpha endosulfan (81.19), lambda cyhalothrin (79.31), ediphenphos (75.57), lindane (74.43), deltamethrin (69.52) and dimethoate (66.05) with RSD <20 % .

The experiment was repeated on three consecutive days and results are represented in Table 20. The reproducibility of eleven pesticides was proved for Acetone extraction method at 0.2,0.1 and 0.05 ppm levels. At 0.2, 0.1 and 0.05 ppm the reproducibility of the pesticides could be proved satisfactory except for hexaconazole at 0.05ppm level.

Results of method validation studies for residue analysis of 2,4-D in soil are presented in Table 21.The mean per cent recovery of 2,4-D was recorded as 92.05, 85.51 and 81.25 per cent at 0.2, 0.1and 0.05 ppm respectively. The reproducibility of this method at three levels was also proved. The mean recoveries were 90.81, 82.46 and 79.47 per cent at 0.2, 0.1 and 0.05 ppm respectively when analyzed at three consecutive days with RSD < 20 %.

Table 21. Recovery of 2,4-D in soil following the acetone extraction method

Level of fortification	Mean Per cent Recovery	SD	RSDr
Repeatability			
0.2 ppm	92.05	2.721	2.957
0.1ppm	85.51	5.246	6.134
0.05ppm	81.25	2.403	2.958
Reproducibility			
Level of fortification	Mean Per cent Recovery	SD	RSD*r*
0.2ppm	90.817	0.758	0.834
0.1ppm	82.46	4.219	5.117
0.05ppm	79.47	2.929	3.685

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r - Relative standard deviation for reproducibility.

4.2.3 Validation of MRM for pesticide residue analysis of paddy grains

Data presented in Table 22 represent the mean recovery for the eleven selected pesticides at three different fortification levels of 0.2, 0.1 and 0.05 ppm. The mean per cent recovery of pesticides in the descending order was chlorpyriphos (92.95), phorate (89.87) , alpha endosulfan (85.42), methyl parathion (80.46) malathion (79.55) , lindane (73.87),lambdacyhalothrin (71.91), ediphenphos (71.76), deltamethrin (68.11), dimethoate(65.17) and hexaconazole (56.75) at 0.2 ppm level with RSD <20

At 0.1 ppm level the recovery of nine pesticides were satisfactory and ranged from 63.69 to 94.89 per cent. The mean per cent recovery in the descending order was chlorpyriphos (94.89), lindane(86.17), methyl parathion (82.88), alpha endosulfan(80.13), deltamethrin(79.55), phorate (71.61), malathion (68.54), lambdacyhalothrin (67.46) and dimethoate (63.69) with RSD <20 % . The recovery was not satisfactory for hexaconazole and ediphenphos at 0.1 ppm.

The repeatability of the nine pesticides were also proved at 0.05 ppm level. The mean recovery ranged from 55.55 to 98.70 per cent. The mean per cent recovery in the descending order was chlorpyriphos (98.70), phorate (94.01), lindane (91.59), methyl parathion (86.39), alpha endosulfan (86.34), malathion (83.00), deltamethrin (80.52), lambda cyhalothrin (74.23) and dimethoate (55.55) with RSD <20 % . The recovery was not satisfactory for hexaconazole and ediphenphos at 0.05 ppm.

The experiment was repeated on three consecutive days and results are represented in Table 23. The reproducibility of eleven pesticides was proved for paddy grains at 0.2 ppm level. At 0.1 ppm and 0.05 ppm the reproducibility of nine pesticides could be proved satisfactorily except for hexaconazole and ediphenphos.

4.2.3 Validation of MRM for pesticide residue analysis of paddy straw

Data presented in Table 24 represent the mean recovery for the eleven selected pesticides at three different fortification levels of 0.2, 0.1 and 0.05 ppm. The mean recovery ranged from 53.19 to 97.55 per cent at 0.2 ppm level. At 0.2 ppm level the mean per cent recovery of pesticides in the descending order was lindane (97.55), chlorpyriphos (97.42), , alpha endosulfan (96.07), dimethoate(82.41), phorate (77.21) malathion,(70.94), methyl parathion (70.13), deltamethrin (67.38), lambdacyhalothrin (61.31), ediphenphos (60.45), and hexaconazole (53.19) at 0.2 ppm level with RSD <20%.

At 0.1 ppm level the mean recovery of pesticides in the descending order was lindane (101.15), chlorpyriphos (98.67), alpha endosulfan (91.14), malathion,(70.98), methyl parathion (70.28), deltamethrin (65.97), lambdacyhalothrin (66.23), phorate (63.14), and

Table 22. Repeatability of MRM of pesticides at three levels in paddy grains.

Pesticides	Level of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean recovery %	SD	RSDr	Mean recovery %	SD	RSDr	Mean recovery %	SD	RSDr
Phorate	89.87	6.528	7.263	71.61	3.686	5.148	94.01	8.490	9.031
Dimethoate	65.17	6.056	9.293	63.69	2.515	3.949	55.55	10.369	18.664
Lindane	73.87	5.752	7.786	86.17	9.398	10.907	91.59	9.055	9.887
Methyl parathion	80.46	1.247	1.550	82.88	6.169	7.444	86.39	6.255	7.240
Malathion	79.55	3.136	3.941	68.54	3.912	5.708	83.00	8.743	10.534
Chlorpyriphos	92.95	3.204	3.447	94.89	5.747	6.056	98.70	1.226	1.242
Alpha endosulfan	85.42	5.798	6.788	80.13	3.878	4.839	86.34	3.360	3.892
Hexaconazole	56.75	10.008	17.637	NS	NS	NS	NS	NS	NS
Ediphenphos	71.76	2.545	3.546	NS	NS	NS	NS	NS	NS
Lambda cyhalothrin	71.91	0.816	1.135	67.46	4.656	6.902	74.23	5.791	7.801
Deltamethrin	68.11	2.657	3.902	79.55	3.512	4.415	80.52	3.456	4.292

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r Relative standard deviation for reproducibility.

Table 23. Reproducibility of MRM of pesticides at three levels in paddy grains

Pesticides	Levels of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean recovery%	SD	RSDr	Mean recovery%	SD	RSDr	Mean recovery%	SD	RSDr
Phorate	88.29	3.791	4.294	73.22	5.006	6.837	99.30	5.595	5.635
Dimethoate	65.17	6.056	9.293	65.89	2.843	4.315	59.12	10.553	17.852
Lindane	76.49	10.855	14.192	87.22	3.282	3.763	96.48	3.302	3.422
Methyl parathion	81.13	1.626	2.004	82.88	6.169	7.444	86.17	2.673	3.102
Malathion	81.49	2.262	2.775	68.35	5.515	8.069	81.69	2.965	3.630
Chlorpyriphos	94.72	1.197	1.264	95.35	5.171	5.423	99.73	2.649	2.656
Alpha endosulfan	85.25	4.560	5.349	83.18	2.252	2.708	85.64	5.264	6.147
Hexaconazole	59.87	10.848	18.119	NS	NS	NS	NS	NS	NS
Ediphenphos	73.51	2.397	3.261	NS	NS	NS	NS	NS	NS
Lambda cyhalothrin	73.46	2.029	2.762	67.45	2.013	2.984	75.52	5.855	7.753
Deltamethrin	69.28	3.627	5.236	82.63	2.428	2.939	85.40	2.758	3.229

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r Relative standard deviation for reproducibility

dimethoate (60.54) with RSD <20%. The recovery of hexaconazole and ediphenphos were not satisfactory at 0.1ppm.

At 0.05 ppm the mean per cent recoveries were satisfactory for nine pesticides except for hexaconazole and ediphenphos. At 0.1 ppm level the mean per cent recovery of pesticides in the descending order was lindane (88.22), methyl parathion (86.68), phorate (72.31), malathion,(69.45), alpha endosulfan (65.42), chlorpyriphos (64.46), deltamethrin (64.29), lambda cyhalothrin (62.98), and dimethoate (62.23) with RSD <20% .The recovery of hexaconazole and ediphenphos were not satisfactory at 0.1ppm.

The reproducibility of this method for the above eleven pesticides in paddy straw was proved at 0.2 ppm, while for nine pesticides except for ediphenphos and hexaconazole , reproducibility was proved at 0.1 ppm and 0.05 ppm (Table 25).

4.2.4 Validation of MRM of pesticide residue analysis of fish

Data presented in Table 26 represent the mean recovery for the eleven selected pesticides at three different fortification levels of 0.2, 0.1 and 0.05 ppm. The mean recovery ranged from 66.33 to 99.74 per cent at 0.2 ppm.

At 0.2 ppm level the mean recovery of pesticides in the descending order was alpha endosulfan (99.74), chlorpyriphos (97.72), lindane (92.15), phorate (90.66) malathion, (89.76), methyl parathion (82.84), lambda cyhalothrin (82.92), deltamethrin (79.63), ediphenphos

Table 24. Repeatability of MRM of pesticides at three levels in paddy straw

Pesticides	Levels of fortification								
	0.2 ppm			0.1 ppm			0.05 ppm		
	Mean Recovery%	SD	RSDr	Mean Recover y%	SD	RSD	Mean Recover y%	SD	RSDr
Phorate	77.20	6.286	8.143	63.14	2.228	3.529	72.31	4.048	5.599
Dimethoate	82.41	6.078	7.375	60.54	4.821	7.963	62.23	1.996	3.207
Lindane	97.55	3.726	3.819	101.15	1.562	1.544	88.22	1.176	1.333
Methyl parathion	70.13	2.430	3.465	70.28	7.535	10.721	86.68	6.073	7.006
Malathion	70.94	1.250	1.763	70.98	1.638	2.309	69.45	2.578	3.712
Chlorpyriphos	97.42	4.027	4.134	98.67	2.664	2.699	64.46	0.404	0.627
Alpha endosulfan	96.07	3.567	3.713	91.14	8.016	8.796	65.42	3.709	5.669
Hexaconazole	53.19	2.003	3.765	NS	NS	NS	NS	NS	NS
Ediphenphos	60.45	0.831	1.375	NS	NS	NS	NS	NS	NS
Lambda cyhalothrin	61.31	0.178	0.290	66.23	4.334	6.545	62.98	5.144	8.169
Deltamethrin	67.38	4.457	6.614	65.97	4.530	6.867	64.29	1.259	1.958

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditio ns on three consecutive days:

RSD r Relative standard deviation for reproducibility

Table 25. Reproducibility of MRM of pesticides at three levels in paddy straw

Pesticides	Levels of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean Recovery %	SD	RSDr	Mean Recover y%	SD	RSDr	Mean Recover y%	SD	RSDr
Phorate	75.57	1.0575	1.399	62.48	2.893	4.631	72.31	4.048	5.599
Dimethoate	79.32	2.900	3.656	62.04	2.871	4.629	62.23	1.996	3.207
Lindane	97.34	3.416	3.509	98.97	1.077	1.088	88.22	1.176	1.333
Methyl parathion	61.69	1.319	2.139	70.28	7.535	10.721	86.68	6.073	7.006
Malathion	73.31	1.039	1.418	70.98	1.638	2.3090	69.45	2.578	3.712
Chlorpyriphos	90.01	8.258	9.174	98.67	2.664	2.699	97.90	0.670	0.684
Alpha endosulfan	97.88	2.769	2.828	91.14	8.016	8.796	90.18	7.552	8.375
Hexaconazole	52.21	1.342	2.570	NS	NS	NS	NS	NS	NS
Ediphenphos	62.45	0.926	1.483	NS	NS	NS	NS	NS	NS
Lambda cyhalothrin	63.24	2.525	3.992	66.23	4.334	6.545	74.57	3.417	4.584
Deltamethrin	69.05	3.312	4.797	65.97	4.530	6.867	67.92	2.170	3.195

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditio ns on three consecutive days:

RSD r Relative standard deviation for reproducibility

(72.49), hexaconazole (71.82) and dimethoate (66.33), at 0.2 ppm level with RSD <20 % .

At 0.1 ppm level the mean per cent recovery of pesticides in the descending order was phorate (103.00) chlorpyriphos (99.98), lindane (86.13), alpha endosulfan (82.34), methyl parathion (73.88), malathion, (72.46), lambda cyhalothrin (69.93), deltamethrin (68.24), ediphenphos (64.05), and dimethoate(62.76), at 0.1 ppm level with RSD <20 %. The recovery of hexaconazole was not satisfactory at 0.1 ppm.

At 0.05 ppm level the mean per cent recovery of pesticides in the descending order was chlorpyriphos (93.18), phorate (88.51), lindane (84.61), malathion (76.21), methyl parathion (72.56), alpha endosulfan (68.89), lambda cyhalothrin (65.33), deltamethrin (63.72), dimethoate(65.34), and ediphenphos (59.44) at 0.05 ppm level with RSD <20 %. The recovery of hexaconazole was not satisfactory at 0.1 ppm.

The results of reproducibility of validation studies on recovery of pesticides in fish are presented in Table 27.The reproducibility of the method was proved for all the eleven pesticides at 0.2 ppm. At 0.1 and 0.05 ppm reproducibility of ten pesticides could only be proved except for hexaconazole.

Table 26. Repeatability of MRM of pesticides at three levels in fish

Pesticides	Levels of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean Recovery%	SD	RSDr	Mean Recovery %	SD	RSD	Mean Recovery %	SD	RSDr
Phorate	90.66	2.647	2.920	103.00	4.290	4.165	88.51	3.165	3.576
Dimethoate	66.33	5.403	8.145	62.76	2.418	3.853	65.34	3.316	5.075
Lindane	92.15	0.742	0.806	86.13	4.042	4.693	84.61	2.656	3.139
Methyl parathion	82.84	6.481	7.825	73.88	1.713	2.319	72.56	3.570	4.921
Malathion	89.76	3.666	4.084	72.46	0.851	1.175	76.21	4.981	6.536
Chlorpyriphos	97.72	3.764	3.852	99.98	4.652	4.653	93.18	5.981	6.418
Alpha endosulfan	99.74	3.196	3.204	82.34	1.893	2.299	68.89	3.387	4.917
Hexaconazole	71.82	2.823	3.930	NS	NS	NS	NS	NS	NS
Ediphenphos	72.49	4.231	5.837	64.05	1.549	2.419	59.44	6.900	11.610
Lambda cyhalothrin	82.92	0.434	0.524	69.93	5.142	7.353	65.33	2.688	4.114
Deltamethrin	79.63	1.325	1.664	68.24	1.526	2.237	63.72	0.910	1.429

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r Relative standard deviation for reproducibility

Table 27. Reproducibility of MRM of pesticides at three levels in fish

pesticides	Levels of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean Recovery%	SD	RSDr	Mean Recovery %	SD	RSD	Mean Recovery %	SD	RSDr
Phorate	94.47	4.793	5.074	102.60	2.917	2.843	90.38	3.352	3.709
Dimethoate	67.49	2.011	2.980	63.72	1.640	2.573	69.12	1.738	2.515
Lindane	93.53	5.302	5.670	85.86	2.114	2.463	85.20	1.313	1.541
Methyl parathion	84.82	2.763	3.257	74.76	1.238	1.657	73.89	1.061	1.436
Malathion	88.29	4.813	5.451	73.23	1.839	2.511	79.25	3.406	4.299
Chlorpyriphos	96.28	3.132	3.253	100.27	0.693	0.691	91.84	6.749	7.349
Alpha endosulfan	90.58	3.062	3.381	84.07	2.735	3.254	74.82	4.598	6.145
Hexaconazole	76.01	2.090	2.750	NS	NS	NS	NS	NS	NS
Ediphenphos	73.72	1.504	2.041	64.76	1.736	2.680	62.55	4.454	7.120
Lambda cyhalothrin	82.94	1.928	2.324	71.83	6.304	8.777	62.32	1.863	2.990
Deltamethrin	80.87	0.722	0.893	68.57	2.364	3.448	60.74	0.552	0.909

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r Relative standard deviation for reproducibility

4.2.5 Validation of MRM of pesticide residue analysis of duck meat

Data presented in Table 28 represent the mean recovery for the eleven selected pesticides at three different fortification levels of 0.2, 0.1 and 0.05 ppm. The mean recovery was in the range of 62.80 to 111.19 at 0.2 ppm level.

At 0.2 ppm level the mean recovery of pesticides in the descending order was chlorpyriphos (111.19), phorate (105.62), lambda cyhalothrin (102.75), lindane (101.22), alpha endosulfan (98.24), methyl parathion (85.75), malathion,(80.74), ediphenphos (72.05), hexaconazole (72.56), deltamethrin (71.82), and dimethoate (62.80) at 0.2 ppm level with RSD <20 % .

At 0.1 ppm level the mean per cent recovery of pesticides in the descending order was chlorpyriphos (112.42), phorate (103.78), lindane (91.49), malathion, (78.52), alpha endosulfan (75.28), dimethoate(70.85), methyl parathion (69.21), lambda cyhalothrin (66.60), deltamethrin (64.34), and ediphenphos (59.88), with RSD <20 %. Hexaconazole residues were recovered satisfactorily at 0.1 ppm.

At 0.05 ppm level the mean recovery of pesticides in the descending order was chlorpyriphos (101.72), lindane (86.16), phorate (88.11), methyl parathion (74.87), alpha endosulfan (74.28), malathion, (71.17), dimethoate (64.85), lambda cyhalothrin (64.60), deltamethrin (63.68), and ediphenphos (55.88), with RSD <20. Hexaconazole residues were recovered satisfactorily at 0.1 ppm.

Table 28. Repeatability of MRM of pesticides at three levels in duck meat

Pesticides	Levels of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean Recovery%	SD	RSDr	Mean Recover y%	SD	RSD	Mean Recover y%	SD	RSDr
Phorate	105.62	7.128	6.749	103.78	4.370	4.210	88.11	5.900	6.696
Dimethoate	62.80	3.398	5.412	70.85	5.415	7.643	64.85	1.428	2.202
Lindane	101.22	10.415	10.290	91.49	0.954	1.042	86.16	3.308	3.839
Methyl parathion	85.75	8.825	10.292	69.21	4.592	6.636	74.87	5.760	7.692
Malathion	80.74	10.317	12.778	78.52	0.957	1.219	71.17	4.782	6.719
Chlorpyriphos	111.19	6.777	6.095	112.42	1.908	1.697	101.72	3.486	3.427
Alpha endosulfan	98.24	9.851	10.028	75.28	2.786	3.701	74.28	5.442	7.326
Hexaconazole	72.56	4.887	6.736	0.00	0.000	NS	0.00	0.000	NS
Ediphenphos	72.05	2.655	3.685	59.88	2.510	4.191	55.88	3.687	6.599
Lambda cyhalothrin	102.75	4.178	4.066	66.60	7.472	11.220	64.60	3.880	6.007
Deltamethrin	71.82	0.685	0.954	64.34	5.654	8.788	63.68	2.522	3.961

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD r Relative standard deviation for reproducibility

Table 29. Reproducibility of MRM of pesticides at three levels in duck meat

Pesticides	Levels of fortification								
	0.2ppm			0.1 ppm			0.05 ppm		
	Mean Recovery %	SD	RSDr	Mean Recovery %	SD	RSD	Mean Recovery %	SD	RSDr
Phorate	109.44	5.478	5.005	99.80	1.263	1.265	88.86	8.791	9.893
Dimethoate	64.41	2.550	3.959	65.27	65.513	0.456	66.43	2.224	3.348
Lindane	99.07	1.677	1.692	92.34	2.225	2.409	90.44	1.336	1.477
Methyl parathion	89.62	3.053	3.406	79.10	3.498	4.423	75.23	3.668	4.876
Malathion	84.53	2.504	2.962	82.25	5.700	6.930	73.25	5.857	7.996
Chlorpyriphos	105.35	5.278	5.009	102.60	2.391	2.331	102.33	0.111	0.109
Alpha endosulfan	103.28	6.933	6.712	77.44	1.728	2.231	79.12	3.272	4.135
Hexaconazole	72.66	0.682	0.939	0.00	0.000	NS	0.00	0.000	NS
Ediphenphos	72.02	1.154	1.602	60.37	4.250	7.041	60.80	4.658	7.661
Lambda cyhalothrin	74.26	4.927	6.634	73.04	2.534	3.469	68.82	2.102	3.054
Deltamethrin	70.72	0.300	0.425	64.78	4.458	6.883	64.60	2.227	3.447

Number of replicates at each level (n)=6 (three extractions with two injections each) Analysis made under the same conditions on three consecutive days:

RSD *r* Relative standard deviation for reproducibility

The reproducibility of the method for all the above pesticides in meat was proved at 0.2 ppm (Table 29). At 0.1 ppm and 0.05 ppm reproducibility was proved for ten pesticides except hexaconazole.

Table 30, presents the limit of detection, linearity regression equation, limit of detection and quantification of different pesticides in different matrices viz., paddy water, paddy soil, paddy grains, paddy straw, and fish and duck meat. The limit of detection ranged from 0.001 µg g^{-1} to 0.02 µg g^{-1}. The r-value ranged from 0.990 to 0.999.The limit of quantification (LOQ) for different pesticides in water ranged from 0.0001 to 0.01 µg g^{-1} while in soil, the limit of quantification ranged from 0.01 to 0.1 µg g^{-1} . The LOQ of different pesticides under study ranged from 0.01 to 0.2 µg g^{-1} in paddy grains, paddy straw, fish and duck meat.

4.2.6 Recovery experiments on MRM for eggs

The recovery studies were conducted for eggs for the selected pesticides at 0.2 ppm. The mean per cent recovery of phorate, dimethoate, methylparathion, malathion, chlorpyriphos, lindane, alphaendosulfan, hexaconazole,ediphenphos,lambdacyhalothrin and deltamethrin at 0.2 ppm level were 72.60, 63.20, 86.50, 82.10, 75.20, 80.60, 76.20, 66.10, 72.90, 73.60 and 72.20 respectively (Table 31).

Table 30. LOD, Linearity and LOQ of pesticides in different matrices under study

Pesticides	RT	LOD (µg g^{-1})	Linearity Regression equation	R^2 value	RSD	LOQ (µg g^{-1})					
						water	soil	paddy grains	Paddy straw	fish	duck meat
Phorate	5.735	0.001	Y=51807.46 X + 563.03	0.999	3.919	0.0001	0.01	0.01	0.01	0.01	0.01
Dimethoate	6.058	0.01	Y=156955.0 X - 2090.85	0.996	3.735	0.01	0.05	0.05	0.05	0.05	0.05
Lindane	6.918	0.001	Y=812956.6 X -28334.33	0.996	8.892	0.0001	0.01	0.01	0.01	0.01	0.01
Methyl parathion	8.929	0.01	Y=401783.5 X + 4397.11	0.999	5.038	0.001	0.05	0.01	0.01	0.01	0.01
Malathion	10.558	0.01	Y=278937.5 X - 4602.41	0.990	6.683	0.001	0.01	0.01	0.01	0.01	0.01
Chlorpyriphos	11.056	0.001	Y=446114.3 X – 388.45	0.998	2.663	0.001	0.01	0.01	0.01	0.01	0.01
Alpha endosulfan	14.376	0.001	Y=674749.6 X -15768.85	0.993	5.256	0.001	0.01	0.01	0.01	0.01	0.01
Hexaconazole	15.264	0.02	Y=460180.7 X -16403.59	0.985	8.561	0.01	0.1	0.2	0.2	0.2	0.2
Ediphenphos	19.193	0.01	Y=129723.4 X – 832.00	0.992	20.820	0.01	0.05	0.2	0.2	0.05	0.05
Lambda cyhalothrin	26.201	0.01	Y=317015.46 X - 10665.95	0.994	8.934	0.01	0.05	0.05	0.05	0.05	0.05
Deltamethrin	35.739	0.01	Y=217719.00 X - 5629.068	0.995	9.340	0.01	0.05	0.05	0.05	0.05	0.05

LOD- Limit of detection, LOQ-limit of quantification, RT- Retension time, - R^2 Regression coeficient

4.2.7 Recovery experiments on acephate, quinalphos and triazophos.

The mean per cent recovery of acephate, monocrotophos and triasophos in different components in rice ecosystem are presented in Table 32. The mean per cent recovery of acephate at 0.2 ppm were 70.36, 82.23, 76.86, 73.55, 78.26 , 76.66 and 70.95 respectively for water, soil, paddy grains, paddy straw, fish, duck meat and duck eggs. The corresponding values for monocrotophos at 0.2 ppm were 73.86, ,74.56, 78.32 , 80.41,75.38, 73.44 and 77.30 respectively for water, soil, paddy grains, paddy straw, fish, duck meat and duck eggs. The mean per cent recovery of triazophos at 0.2 ppm were 92.86 , 86.55, 95.46, 92.88, 86.86 and 93.25 respectively for water, soil, paddy grains, paddy straw, fish, duck meat and duck eggs.

4.3 Monitoring of pesticide residues in abiotic and biotic components of Kuttanadu ecosystem

The samples of water, soil, paddy grains, paddy straw, fish ,mollusk, duck meat, eggs and beef were collected during the puncha season of 2007-2008 from the three catchments under study and were analyzed for the presence of pesticide residues.

Table 31 Recovery of different pesticides in duck eggs following MRM (Sharma,2007)

Pesticides	Mean recovery (%)	SD	RSD
Phorate	72.60	3.665	5.048
Dimethoate	63.23	1.235	1.954
Lindane	86.50	5.178	5.986
Methyl parathion	82.15	3.42	4.166
Malathion	75.20	1.866	2.481
Chlorpyriphos	80.62	1.993	2.473
Alpha endosulfan	76.22	1.325	1.739
Hexaconazole	66.16	6.445	9.750
Ediphenphos	72.95	3.115	4.273
Lambda cyhalothrin	73.60	2.186	2.970
Deltamethrin	72.23	5.433	7.525

SD: Standard deviation RSD: Relative standard deviation

Table 32 Recovery of acephate, quinalphos and monocrtophos following MRM

Pesticides	**RT**	**Recovery per cent**						
		water	soil	paddy grains	paddy straw	fish	duck meat	Duck eggs
Acephate	5.753	70.36	82.23	76.86	73.55	78.26	76.66	70.95
Monocrotophos	8.513	73.86	74.56	78.32	80.41	75.38	73.44	77.30
Triazophos	15.701	92.86	92.66	86.55	95.46	92.88	86.86	93.25

RT: Retention Time

4.3.1 Monitoring of pesticide residues from catchment I: Catchment of Pampa

Samples were collected from two padashekarams *viz*. Edathua and Thakazhy of catchment of Pampa. Samples of water and soil were collected at different growth stages of rice *viz*., seedling stage, tillering stage, booting stage and milky stage. The samples of paddy grain, paddy straw, fish, mollusk ,duck meat,eggs and beef were collected and analysed.

4.3.1.1 Padashekharam in Edathua

The data on residues of pesticides in samples of water, soil/sediment collected at seedling, tillering booting and milky stage of rice and from samples of fish, mollusk, duck meat,eggs and beef collected after harvest of the crop from the padashekharam in Edathua are presented in Table 33.

Data presented on 33 revealed that residues of herbicide alone was detected in the water samples collected at the seedling stage from paddy field, outlet and drainage channel. Out of the three water samples collected from the paddy field of padashekaram at the seedling stage of the crop, two samples showed the presence of residues of 2-4 D, the level being 0.052 to 0.054ppm. Out of the three water samples collected from the field outlets, 2,4-D residues were detected in two samples both at the level of 0.02 ppm. Among the three

Table 33 Residues of pesticides in biotic and abiotic components in Catchment of Pampa :Location : Edathua

Component	Water					Soil/ sediment				
Stage of sample collection	Paddy field	Field outlet	Drainage channel	Stream	River	Paddy field	Field outlet	Drainage channel	Stream	River
Seedling stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	2	2	1	BDL	BDL	2	1	BDL	BDL	BDL
Pesticides detected	a)2,4-D	a)2,4-D	a)2,4-D	BDL	BDL	a) Dimethoate b) 2,4-D	a)2,4-D	BDL	BDL	BDL
Mean residues in ppm	0.052 0.054	0.02 0.02	0.01	BDL	BDL	a)0.3 ppm b)0.03 ppm	a)0.02	BDL	BDL	BDL
Tillering stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	2	1	Nil	Nil	Nil	Nil	Nil	BDL	BDL	BDL
Pesticides detected	MCP	MCP	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean residues in ppm	0.025 0.030	0.01	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Booting										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Milky stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

After Harvest							
Component	Grain	Straw	Duck meat	Eggs	Fish	Mollusk	Beef
Analysed	6	6	6	6	6	6	6
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL

water samples collected from the adjoining drainage channel, one showed the presence of 2,4- D residues at the level of 0.01 ppm. No detectable residue was present in water samples collected either from the stream or the river at seedling stage.

Among the water samples collected from the paddy fields at tillering stage of the crop, two of them showed the presence of residues of monocrotophos to the tune of 0.025 ppm and 0.03 ppm. One sample from field outlet also showed monocrotophos residues at a level of 0.01 ppm. As in the case of sampling at tillering stage, there were no detectable residues in samples collected either from the stream or in river. At the booting and milky stage, irrespective of the site of collection, all the samples of water at were free of residues.

The data on residues of pesticides in soil samples collected at the seedling stage from the padashekharam in Edathua indicated that out of the three soil samples collected from the paddy field, one showed the presence of dimethoate at a levelof 0.3 ppm and another with 2,4-D to the tune of 0.03 ppm. Of the three samples collected from the field outlet, one sample indicated the presence of 2,4-D to the tune of 0.02 ppm. All the samples of soil / sediment collected from drainage channel, stream or river were found free of residues. Similarly, all the soil samples collected at tillering, booting and milky stage were also found free of pesticide residues.

The data on residues of pesticides in samples of paddy grains and straw collected at harvest from the padashekharam in Edathua indicated that all the samples were free from pesticide residues.

The data on residues of pesticides in samples of fish, mollusk,duck meat,eggs and beef collected after harvest from the padashekharam in Edathua indicated that all the samples were free from pesticide residues.

4.3.1.2. Padashekharam in Thakazhy

The data on residues of pesticides in samples of water, soil/sediment collected at seedling, tillering, booting and milky stage of rice and from samples of fish, mollusk, duck meat, eggs and beef collected after harvest from the padashekharam in Thakazhy are presented in Table 34

Data revealed that residues of herbicide alone was detected in the water samples collected at the seedling stage. Out of the three water samples collected from the paddy field at the seedling stage of the crop, one sample alone showed the presence of residues of 2-4 D, the level being 0.36 ppm. Out of the three water samples collected from the field outlets also only one sample showed the presence of residues of 2,4-D at the level of 0.05 ppm. Among the three water samples collected from the adjoining drainage channel, one showed the presence of 2,4- D residues at level of 0.014 ppm. No detectable

Table 34 Residues of pesticides in biotic and abiotic components in Catchment of Pampa:Location : Thakazhy

Stage of sample collection	**Water**					**Soil/ sediment**				
	Paddy field	**Field outlet**	**Drainage channel**	**Stream**	**River**	**Paddy field**	**Field outlet**	**Drainage channel**	**Stream**	**River**
Seedling stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	1	1	1	BDL	BDL	1	1	1	BDL	BDL
Pesticides detected	a)2,4-D	a)2,4-D	a)2,4-D	BDL	BDL	a)2,4-D	a)2,4-D	a)2,4-D	BDL	BDL
Mean residues in ppm	0.36	0.05	0.014	BDL	BDL	0.2	0.02	0.018	BDL	BDL
Tillering stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pesticides detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean residues in ppm	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Booting										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Milky stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

After Harvest							
	Grain	Straw	Duck meat	Eggs	Fish	Mollusk	Beef
Analysed	6	6	6	6	6	6	6
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL

residue was present in water samples collected either from the stream or the river.

Among the water samples collected from the paddy fields at tillering stage of the crop, none of the samples showed the presence of pesticide residues above the detectable level. Samples collected at the booting and milky stage, were also free of pesticide residues.

The data on residues of pesticides in soil samples collected at the seedling stage from the padashekharam in Thakazhy revealed that out of the three soil samples collected from the paddy field, one showed the presence of 2,4-D to the tune of 0.20 ppm. Of the three samples collected from the field outlet, one sample indicated the presence of 2,4-D to the tune of 0.02 ppm. Similarly, one samples collected from drainage channel also showed the presence of 2,4-D residues at 0.018 ppm level. All the samples of soil / sediment collected from stream or river were found free of residues. All the soil samples collected at tillering stage, booting stage and milky stage were also found free of pesticide residues.

The data on residues of pesticides in samples of paddy grains and straw collected at harvest from the padashekharam in Thakazhy indicated that all the samples were free from pesticide residues.

The data on residues of pesticides in samples of fish,mollusk, duck meat,eggs and beef collected after harvest from the padashekharam in Thakazhy indicated that all the samples were free from pesticide residues.

4.3.2 Monitoring of pesticide residues from catchment II: Catchment of Manimala

Samples were collected from two padashekarams *viz*. Kidangara and Ramankari of catchment of Manimala. Samples of water and soil were collected at four different growth stages of rice *viz*., seedling stage, tillering stage, booting stage and milky stage. The samples of grain, straw, fish, mollusk,duck meat,eggs and beef were collected and analysed after harvest.

4.3.2.1 Padashekharam in Kidangara

The data on residues of pesticides in samples of water, soil/sediment collected at seedling, tillering, booting and milky stage of rice and from samples of fish,mollusk, duck meat,eggs and beef collected after harvest from the padashekharam in Kidangara are presented in Table 33.

Data revealed that residues of herbicide alone were detected in the water samples collected at the seedling stage. All the three water samples collected from the padashekaram at the seedling stage of the crop, showed the presence of residues of 2-4 D, the level being 0.025 , 0.043 and 0.056 ppm. Out of the three water samples collected from the field outlets, 2,4-D residue was detected in one sample at the level of

Table 35 Residues of pesticides in biotic and abiotic components in Catchment of Manimala:Location : Kidangara

Stage of sample collection	**Water**					**Soil/ sediment**				
	Paddy field	**Field outlet**	**Drainage channel**	**Stream**	**River**	**Paddy field**	**Field outlet**	**Drainage channel**	**Stream**	**River**
Seedling stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	3	1	1	BDL	BDL	1	1	BDL	BDL	BDL
Pesticides detected	a)2,4-D	a)2,4-D	a)2,4-D	BDL	BDL	a)2,4-D	a)2,4-D	BDL	BDL	BDL
Mean residues in ppm	0.025, 0.056, 0.043	0.02	0.01	BDL	BDL	0.02	0.01	BDL	BDL	BDL
Tillering stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	1	1	1	BDL	BDL	1	1	1	BDL	BDL
Pesticides detected	chlorpyri phos	chlorpyri phos	chlorpyrip hos	BDL	BDL	chlorpyri phos	chlorpyri phos	chlorpyrip hos	BDL	BDL
Mean residues in ppm	0.02	0.01	0.005	BDL	BDL	0.32	0.26	0.01	BDL	BDL
Booting										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Milky stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
After Harvest										

	Grain	**Straw**	**Duck meat**	**Eggs**	**Fish**	**Mollusk**	**Beef**
Analysed	6	6	6	6	6	6	6
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL

0.02 ppm. Among the three water samples collected from the adjoining drainage channel, one showed the presence of 2,4- D residues at very low level of 0.01 ppm. No detectable residue was present in water samples collected either from the stream or the river.

Among the water samples collected from the paddy fields at tillering stage of the crop, one of them showed the presence of residues of chlorpyriphos to the tune of 0.02 ppm. One sample from field outlet also showed residues of chlorpyriphos at a level of 0.01 ppm. Chlorpyriphos residues were also detected in samples collected from the drainage channel at the level of 0.005 ppm. No detectable residue was present in water samples collected either from the stream or the river. At the booting and milky stage, irrespective of the site of collection, all the samples of water were free of residues.

The data on residues of pesticides in soil samples collected at the seedling stage from the padashekharam in Kidangara indicated that out of the three soil samples collected from the paddy field, one showed the presence of 2,4-D at a level of 0.02 ppm. Of the three samples collected from the field outlet, one sample indicated the presence of 2, 4-D to the tune of 0.01 ppm. Of the three samples collected from the drainage channel, one sample indicated the presence of 2, 4-D as traces. All the samples of soil / sediment collected from stream or river were found free of residues.

The data on residues of pesticides in soil samples collected at the tillering stage from the padashekharam in Kidangara indicated that out of the three soil samples collected from the paddy field, one showed the presence of chlorpyriphos at a levelof 0.32 ppm. Of the three samples collected from the field outlet, one sample indicated the presence of chlorpyriphos to the tune of 0.26 ppm. Of the three samples collected from the drainage channel, one sample indicated the presence of chlorpyriphos to the tune of 0.01 ppm. All the samples of soil / sediment collected from stream or river were found free of residues. All the soil samples collected at booting stage and milky stage were also found free of pesticide residues.

The data on residues of pesticides in samples of paddy grains and straw collected at harvest from the padashekharam in Kidangara indicated that all the samples were free from pesticide residues.

The data on residues of pesticides in samples of fish, mollusk,duck meat,eggs and beef collected after harvest from the padashekharam in Kidangara indicated that all the samples were free from pesticide residues.

4.3.2.2 Padashekharam in Ramankari

The data on residues of pesticides in samples of water, soil/sediment collected at seedling, tillering, booting and milky stage of rice and from samples of fish, mollusk, duck meat,eggs and beef collected after harvest from the padashekharam in Ramankari are presented in Table 34.

Data revealed that residues of herbicide alone were detected in the water samples collected at the seedling stage. Of the three water samples collected from the padashekaram at the seedling stage of the crop, two of them showed the presence of residues of 2-4 D, the level being 0.038 and 0.035 ppm. Out of the three water samples collected from the field outlets, 2,4-D residue was detected in one sample at the level of 0.02 ppm. No detectable residue was present in water samples collected either from the adjoining drainage channel or the stream or the river.

Among the water samples collected from the paddy fields at tillering stage of the crop, one of them showed the presence of residues of triazophos to the tune of 0.03 ppm. One sample from field outlet also showed residues of triazophos at a level of 0.02 ppm. Triazophos residues was also detected in samples colected from the drainage channel at the level of 0.01 ppm. No detectable residue was present in water samples collected either from the stream or the river.

At the booting of the crop, one each of the water samples collected from paddy field and field outlet indicated the presence of malathion residues to the level of 0.1ppm and 0.05 ppm respectively. The sample from drainage channel, river and stream collected at the booting stage were free of residues

Table 36 Residues of pesticides in biotic and abiotic components of Catchment of Manimala: Location : Ramankari

Stage of sample collection	Water					Soil/ sediment				
	Paddy field	Field outlet	Drainage channel	Stream	River	Paddy field	Field outlet	Drainage channel	Stream	River
Seedling stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	2	1	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pesticides detected	a)2,4-D	a)2,4-D	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean residues in ppm	0.038, 0.035	0.02	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Tillering stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	1	1	1	BDL	BDL	2	1	1	BDL	BDL
Pesticides detected	Triazophos	Triazophos	Triazophos	BDL	BDL	1.Triazophos 2.Quinalphos	Triazophos	Triazophos	BDL	BDL
Mean residues in ppm	0.03	0.02	0.0.01	BDL	BDL	0.32 0.05	0.043	0.02	BDL	BDL
Booting										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	1	1	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pesticide detected	Malathion	Malathion	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean residues in ppm	0.1	0.05	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Milky stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL

After Harvest							
	Grain	**Straw**	**Duck meat**	**Eggs**	**Fish**	**Mollusk**	**Beef**
Analysed	6	6	6	6	6	6	6
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL

Irresepective of the site of collection, all the samples of water analyzed during the milky stage were free of residues.

The data on residues of pesticides in soil samples collected at the seedling stage from the padashekharam in Ramankari indicated that the residues were below the detectable level in all the three samples collected from the paddy field, field outlet, drainage channel, stream and river.

The data on residues of pesticides in soil samples collected at the tillering stage from the padashekharam in Ramankari indicated that out of the three soil samples collected from the paddy field, one showed the presence of both triazophos and quinalphos to the tune of 0.32 and 0.05 ppm respectively. Of the three samples collected from the field outlet, one sample indicated the presence of triazophos to the tune of 0.043 ppm. Of the three samples collected from the drainage channel, one sample indicated the presence of triazophos to the tune of 0.02 ppm. All the samples of soil / sediment collected from the stream or river showed residues below the detectable level.

All the soil samples collected at booting stage and milky stage showed that the residues were below the detectable level.

The data on residues of pesticides in samples of paddy grains and straw collected at harvest from the padashekharam in Ramankari indicated that residues were below the detectable level.

The data on residues of pesticides in samples of fish, mollusk,duck meat,eggs and beef collected after harvest from the padashekharam in Ramankari indicated that residues were below the detectable level.

4.3.3 Monitoring of pesticide residues from catchment III: Catchment of Meenachil

4.3.3.1 Padashekharam in Kumarakom

The data on residues of pesticides in samples of water, soil/sediment collected at seedling, tillering, booting and milky stage of rice and from samples of fish, mollusk, duck meat, eggs and beef collected after harvest from the padashekharam in Kumarakom are presented in Table 37.

Data revealed that residues of herbicide alone was detected in the water samples collected at the seedling stage. Of the three water samples collected from the padashekaram at the seedling stage of the crop, two of them showed the presence of residues of 2-4 D, the level being 0.03 and 0.035 ppm. Out of the three water samples collected from the field outlets, 2,4-D residue was detected in one sample at the level of 0.02 ppm. Water samples collected from the adjoining drainage channel showed residues of 2,4-D to a levelof 0.01 ppm. No detectable residue was present in water samples collected either from the stream or the river.

Table 37 Residues of pesticides in biotic and abiotic components in Catchment of Meenachil:Location : Kumarakom

	Water					**Soil/ sediment**				
	Paddy field	Field outlet	Drainage channel	Stream	River	Paddy field	Field outlet	Drainage channel	Stream	River
Seedling stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	2	1	1	BDL	BDL	1	1	BDL	BDL	BDL
Pesticides detected	a)2,4-D	a)2,4-D	a)2,4-D	BDL	BDL	Dimethoate	Dimethoate	BDL	BDL	BDL
Mean residues in ppm	0.03, 0.035	0.02	0.01	BDL	BDL	0.02	0.01	BDL	BDL	BDL
Tillering stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	1	BDL	BDL	BDL	1.Carbaryl	BDL			
Pesticides detected	BDL	MCP	BDL	BDL	BDL	0.32	BDL	BDL	BDL	BDL
Mean residues in ppm	BDL	0.01	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Booting										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected			BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pesticide detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean residues in ppm	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Milky stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
After Harvest										
	Grain	**Straw**	**Duck meat**	**Eggs**	**Fish**	**Mollusk**	**Beef**			
Analysed	6	6	6	6	6	6	6			
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL			

Though water samples collected from the paddy fields at tillering stage of the crop, did not show any residues, sample collected from the field outlet showed residues of monocrotophos at a level of 0.01 ppm. No detectable residue was present in water samples collected either from the stream or the river. Residues in the sample collected from the drainage channel, river and stream at the booting and milky stages were below the detectable level.

The data on residues of pesticides in soil samples collected at the seedling stage from the padashekharam in Kumarakom indicated the presence of 0.02 ppm of dimethoate in the paddy field and 0.01 ppm in field outlet. The residues were below the detectable level in all the three samples collected from the drainage channel, stream and river.

The data on residues of pesticides in soil samples collected at the tillering stage from the padashekharam in Kumarakom indicated that out of the three soil samples collected from the paddy field, one showed the presence of carbaryl to the tune of 0.32 ppm. All the samples of soil / sediment collected from the field outlet, drainage channel, stream and river showed residues below the detectable level.

All the soil samples collected at booting stage and milky stage showed that the residues were below the detectable level.

The data on residues of pesticides in samples of paddy grains and straw collected at harvest from the padashekharam in Kumarakom indicated that residues were below the detectable level.

The data on residues of pesticides in samples of fish, mollusk,duck meat,eggs and beef collected after harvest from the padashekharam in Kumaakom indicated that residues were below the detectable level.

4.3.3.2 Padashekharam in Thiruvarpu

The data on residues of pesticides in samples of water, soil/sediment collected at seedling, tillering booting and milky stage of rice and from samples of fish, mollusk,duck meat ,eggs and beef collected after harvest from the padashekharam in Thiruvarpu are presented in Table 38.

Data revealed that residues of herbicide alone was detected in the water samples collected at the seedling stage. Of the three water samples collected from the padashekaram at the seedling stage of the crop, two of them showed the presence of residues of 2-4 D, the level being 0.03 and 0.05 ppm. Out of the three water samples collected from the field outlets, 2,4-D residue was detected in one sample at the level of 0.02 ppm. The residues were below the detectable level in water samples collected from adjoining drainage channel, the stream and the river.

Table 38 Residues of pesticides in in biotic and abiotic components in Catchment of Meenachil:Location : Thiruvarpu

	Water					**Soil/ sediment**				
	Paddy field	Field outlet	Drainage channel	Stream	River	Paddy field	Field outlet	Drainage channel	Stream	River
Seedling stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	2	1		BDL	BDL	1	1	BDL	BDL	BDL
Pesticides detected	a)2,4-D	a)2,4-D		BDL	BDL	a)2,4-D	a)2,4-D	BDL	BDL	BDL
Mean residues in ppm	0.03, 0.05	0.02		BDL	BDL	0.03	0.01	BDL	BDL	BDL
Tillering stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	1	1	BDL	BDL	BDL	1	BDL	BDL	BDL	BDL
Pesticides detected	Phorate	Phorate	BDL	BDL	BDL	1.Phorate sulfone 2.	BDL	BDL	BDL	BDL
Mean residues in ppm	0.062	0.038	BDL	BDL	BDL	0.502	BDL	BDL	BDL	BDL
Booting										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Pesticide detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Mean residues in ppm	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Milky stage										
Analysed	3	3	3	3	3	3	3	3	3	3
Detected	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
After Harvest										
	Grain		**Straw**		**Duck meat**	**Eggs**	**Fish**	**Mollusk**	**Beef**	
Analysed	6		6		6	6	6	6	6	
Detected	BDL		BDL		BDL	BDL	BDL	BDL	BDL	

One of the water samples collected from the paddy fields showed residues of phorate to the tune of 0.062 ppm while another sample from the field outlet showed residues of 0.038 ppm of phorate at tillering stage of the crop. The residues in water samples collected from the drainage channel ,stream and the river were below the detectable level.

Residues in all the sample collected from the paddy field, drainage channel, river and stream at the booting and milky stages were below the detectable level.

The data on residues of pesticides in soil samples collected at the seedling stage from the padashekharam in Thiruvarpu indicated the presence of 0.03 ppm of 2,4-D in the paddy field and 0.01 ppm in field outlet in one sample each. The residues were below the detectable level in all the three samples collected from the drainage channel, stream and river.

The data on residues of pesticides in soil samples collected at the tillering stage from the padashekharam in Thiruvarpu indicated that out of the three soil samples collected from the paddy field, one showed the presence of one of the metabolites of phorate *viz.* phorate sulphone to the tune of 0.502 ppm. All the samples of soil / sediment collected from the field outlet, drainage channel, stream and river showed residues below the detectable level.

All the soil samples collected at booting stage and milky stage showed that the residues were below the detectable level. The data on residues of pesticides in samples of paddy grains and straw collected at harvest from the padashekharam in Thiruvarpu indicated that residues were below the detectable level. The data on residues of pesticides in samples of fish, mollusk, duck meat,eggs and beef collected after harvest from the padashekharam in Thiruvarpu indicated that residues were below the detectable level.

4.4. Impact of conventional and IPM practices on major pests, natural enemies and neutrals

The observations were recorded from two padashekarams following IPM and conventional (Non IPM) practices from catchment of river Manimala.

4.4.1.1 Assessment of pest population

The population of major pests in IPM and conventional (non-IPM) plots are presented in Table 39. The data revealed that at 25DAT the mean population of rice leaf roller *C. medinalis* in plots receiving IPM practices was 5.8$\pm$ 0.837 and it was 5.6 $\pm$ 2.191 in plots following conventional methods. At 45 DAT the mean population was of *C. medinalis* were 17.6 $\pm$ 3.847 and 12.8 $\pm$ 3.834 respectively in IPM and non-IPM plots. The mean population of *C. medinalis* at 65 DAT were 14.2 $\pm$ 2.863 and and 9.8 $\pm$ 1.643 in IPM and non-IPM plots.

Table 39. Population of major pests in IPM and non-IPM plots in Kuttanadu

Pest	Mean number					
	25 DAT		45 DAT		65 DAT	
	IPM	Non-IPM	IPM	Non-IPM	IPM	Non-IPM
C.medinalis	5.8	5.6	17.6	12.8	14.2	9.8
SD	0.837	2.191	3.847	3.834	2.863	1.643
Nephotettix sp	11.6	7.6	44.6	28	39.6	33.8
SD	3.209	2.702	9.659	6.465	18.160	5.495
C.spectra	2.6	1.4	4.2	1.8	3.6	2.2
SD	2.191	1.140	2.387	1.304	3.050	0.837
L.acuta adult	-	-	0.8	0.6	5.0	3.4
SD			0.837	0.548	1.871	0.894

SD –Standard deviation   DAS : Days after Sowin

The population of *Nephotettix* spp at 25 DAS was 11.6 ± 3.209 and 7.6 ± 2.702 repectively in IPM and non-IPM plots. At 45 DAT the population of *Nephotettix* spp was 44.6 ± 9.659and 28 ± 6.465 respectively in IPM and non-IPM plots. At 65 DAS the pest population was 39.6 ± 18.160 while in non-IPM the corresponding values was 33.8 ±5.495.

The population of *C. spectra* at 25 DAS was 2.6 ± 2.191 and 1.4 ± 1.140 in IPM and non-IPM, plots respectively. At 45 DAS the population of *C. spectra* was 4.2 ± 2.387 and 1.8 ±1.304 non-IPM plots respectively. The population of *C. spectra* at 65 DAS was 3.6 ± 3.050 and 2.2 ± 0.837 in IPM and non-IPM plots respectively.

L. acuta was observed on 45 DAS was 0.8 ± 0.837 and 0.6± 0.548 respectively in IPM and non IPM plots while at 65 DAS the population was 5.0 ± 1.871 and 3.4 ± 0.894 respectively.

4.4.1.1 Assessment of population of natural enemies and neutrals

The population of natural enemies and neutrals in IPM and non-IPM plots are presented in Table 40.

The mean population of *C. lividipennis* at 25 DAS was 14.0 ± 3.742 and 9.0 ± 1.225 respectively in IPM and non-IPM plots respectively. At 45 DAS the population was 25.60 ± 4.336 and 14.20 ± 3.962 in IPM and non-IPM plots respectively while the values were 5.675 ±2.40 and 9.60 ±1.517 at 65 DAS.

Table 40. Mean population of natural enemies and neutrals in IPM and non- IPM plots in Kuttanadu

Natural enemy	Mean number					
	25 DAT		45 DAT		65 DAT	
	IPM	Non-IPM	IPM	Non-IPM	IPM	Non-IPM
C. lividipennis	14.00	9.00	25.60	14.20	21.80	9.60
SD	3.742	1.225	4.336	3.962	5.675	1.517
Agriocnemis	2.60	2.20	1.60	0.80	2.40	1.40
SD	2.793	1.304	1.517	0.447	1.140	1.140
Dragon flies	1.80	1.60	3.60	1.20	2.40	1.20
SD	1.304	0.548	0.894	0.837	0.894	0.447
Tetragnatha	0.00	0.00	1.80	1.20	1.40	0.60
SD	0.00	0.00	0.837	0.837	1.140	0.548
L.pseudoannulata	0.00	0.00	0.00	0.00	2.00	0.80
SD	0.00	0.00	0.00	0.00	1.225	0.447
Tetrastichus	0.00	0.00	2.60	1.80	3.20	1.60
SD	0.00	0.00	1.817	0.837	1.304	0.548
Telenomus	0.00	0.00	1.40	1.20	2.200	1.00
SD	0.00	0.00	1.517	0.447	2.490	1.225
Neutrals						
Culicids	0.00	0.00	13.60	4.20	16.60	16.40
SD	0.00	0.00	4.393	2.588	5.413	9.397
Chironomids	3.60	2.200	11.20	9.40	16.20	11.80
SD	2.408	1.924	2.280	5.225	3.493	2.864
House flies	0.00	0.00	2.20	0.80	3.40	1.20
SD	0.00	0.00	1.095	0.447	1.517	0.837
Total pests	20.000	14.600	67.200	43.2	62.400	49.200
Total Defenders	19.400	12.800	36.6	20.4	35.4	16.200
P/D ratio	1.030928	1.140625	1.836066	2.117647	1.762712	3.037037

SD –Standard deviation

In case of *Agriocnemis,* the mean population at 25 DAS was 2.60 ± 2.793 and 2.20 ± 1.304 respectively in IPM and non-IPM plots respectively. At 45 DAS the population was 1.60±1.517 and 0.800±0.447 in IPM and non-IPM plots while the values were 1.14 ± 2.40 and 1.140 ± 1.20 at 65 DAS.

The mean population of dragon flies at 25 DAS was 1.80 ±1.304 and 1.60±0.548 in IPM and non-IPM plots respectively. At 45 DAS the population was 3.60± 0.894 and 1.20±0.837in IPM and non-IPM plots while the values were 2.40 ± 0.894 and 1.20 ± 0.447at 65 DAS.

The spider *Tetragnatha* sp was not observed at 25 DAS in the plots under study. At 45 DAS the mean population of *Tetragnatha* sp was 1.80±0.837 and 1.2 ±0.837 while at 65 DAS the mean population was 1.40±1.140 and 0.60±0.548 respectively for IPM and non-IPM plots respectively.

The spider species *L. pseudoannulata* was absent at 25 DAS and 45 DAS in both the IPM and non-IPM plots while the mean population of *L. pseudoannulata* was recorded as 2.00±1.225 and 0.80±0.447 at 65 DAS in the IPM and non-IPM plots respectively

The *Tetrastichus* sp which was absent during 25 DAS was observed at 45 DAS and 65 DAS. The mean population was 2.60±1.817 and 1.80±0.837 at 45 DAS while it was 3.20±1.304 and 1.60±0.548 in IPM and non-IPM plots.

The population of *Telenomus sp.* was also absent at 25 DAS. The mean population was 1.40 ±1.517 and 1.20±0.447 at 45 DAS and 2.20±2.490 and 1.00±1.225 at 65 DAS in IPM and non-IPM plots.

The populations of neutrals present in the rice viz., culicids, chironomids and house flies in the plots were also recorded. The culicids were absent during 25 DAS while mean population at 45 DAS was 13.60±4.393 and 4.20 ±2.588 respectively in IPM and non-IPM plots. At 65 DAS the mean population was 16.60±5.413 and 16.40 ±9.397 respectively in IPM and non-IPM plots.

At 25 DAS the mean population of chironomids was 3.60±2.408 and 2.20 ±1.924 in IPM and non-IPM plots while at 45 DAS the values were 11.20 ± 2.28 and 9.40±5.225 respectively. At 65 DAS the population was 16.20±3.493 and 11.80±2.864 respectively at IPM and non-IPM plots.

The house flies were absent at 25 DAS while the mean population at 45 DAS was 2.20±1.095 and 0.80±0.447 respectively at IPM and non-IPM plots. At 65 DAS the mean population was 3.40±1.517 and 1.20 ± 0.837 respectively at IPM and non-IPM plots.

The pest defender ratio during different intervals was also assessed. At 25DAS the P/D ratio was 1.031 and 1.141 in non-IPM. At 45 DAS, the P/D ratio was 1.836 in IPM and 2.118 non-IPM. At 65 DAS the P/D ratio was 1.763 in IPM plots and 3.037 non-IPM .

Table 41. Mean percent damage of rice stem borer and rice leaf roller in IPM and Non IPM fields

Name of Pest	Per cent damage at intervals					
	25DAT		45 DAT		65DAT	
	IPM	Non-IPM	IPM	Non- IPM	IPM	Non-IPM
Rice Stem borer	2.61	1.73	8.84	4.94	12.95	8.87
SD	1.152	0.400	1.173	0.594	1.847	1.714
Rice leaf Roller	2.07	1.97	2.8	2.5	3.33	2.7
SD	0.153	0.208	0.4	0.360	0.461	0.608

SD –Standard deviation

Table 42. Grain and straw yield in IPM and Non-IPM plots

Yield	Mean weight (t per ha)	
	IPM	Non-IPM
Grain Yield	3.65	3.44
SD	0.312	0.104
Straw Yield	8.0788	7.7873
SD	0.255	0.331

SD –Standard deviation

4.4.2 Damage scoring of major pests

The data on mean damage by two major rice pests viz., rice stem borer *S. incertulus* and *C. medinalis* are presented in Table 39. At 25 DAS the damage score for *S. incertulus* was recorded as 2.61±1.152 in IPM plots while it was 1.73 ±0.400 in non IPM plots. At 45 DAS, the damage score was 8.84±1.173 and 4.94±0.594 respectively at IPM and non-IPM plots.The damage scores were 12.95±1.847 and 8.87 ±1.714 at 65 DAS for IPM and non-IPM plots.

At 25 DAS the damage score recoded for *C. medinalis* was 2.07 ± 0.153 and 1.97 ±0.208 respectively at IPM and non-IPM plots. At 45 DAS the values were 2.8 ±0.400 and 2.5 ±0.360 for IPM and non-IPM plots while the damage score was 3.33±0.461 and 2.7 ±0.608 respectively for IPM and non-IPMat 65 DAS.

4.4.3 Yield in IPM and non-IPM plots

The mean grain yield and straw yield per ha in IPM and non-IPM plots are presented in Table 42. The mean grain yield in IPM plots was 3.65 ± 0.312 while that in non-IPM plot was 3.44 ± 0.104.The mean straw yield was higher in IPM plots (8.08 ± 0.255) than in non-IPM plots (7.79 ± 0.331).

4.4.4 Pesticide residue analysis in food and environmental samples from IPM and non IPM plots.

The data on the residues revealed that the mean level of residues in the paddy grains, straw, meat, fish and soil samples were below the detectable level of 0.01ppm when sampled at the time of harvest from both in the IPM and non IPM plots.

4.5 Dissipation of pesticides in Kuttanadu rice ecosystem

4.5.1 Field situation I

The results of dissipation of the residues of 2,4-D, chlorpyriphos, acephate, hexaconazole and lambda cyhalothrin both in the soil and water following application in the rice field to the adjoining field outlet, drainage channel, stream and finally to the river are presented in tables 43 to 50.

4.5.1.1 Dissipation of 2,4-D in rice soil

Mean residues of 2,4-D in soil observed at different intervals after treatment are presented in Table 43. The data indicated that the initial deposit (at 2 HAS) in soil in paddy field was 3.602 ppm while it was 2.62 ppm in the field outlet. Residues were below the detectable level of 0.01 ppm in the drainage channel. Samples from stream and river also did not show the presence of 2,4-D residues. At 24 HAS, the mean residues were 2.47 and 1.92 ppm respectively in paddy field and field outlet. The samples collected from drainage channel, stream and river did not show the presence of the herbicide residues. At 48 HAS the mean residues of 2,4-D were 2.157 and 1.66 ppm respectively in

Table 43. Residues of 2,4-D in soil at intervals in Kuttanadu rice ecosystem (Field situation-1)

Intervals	Mean residues 2,4D in soil (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	3.602	2.62	BDL	BDL	BDL
24 HAS	2.470	1.92	BDL	BDL	BDL
48 HAS	2.157	1.66	BDL	BDL	BDL
54 HAS	0.883	1.250	BDL	BDL	BDL
72 HAS	0.328	0.825	BDL	BDL	BDL
4 DAS	0.052	0.502	0.02	BDL	BDL
5 DAS	0.017	BDL	BDL	BDL	BDL
7 DAS	BDL	BDL	BDL	BDL	BDL

HAS-Hours after spraying, DAS- Days after spraying, BDL- Below detectable limit

Table 44. Residues of 2,4-D in water at intervals in Kuttanadu rice ecosystem

(Field situation-1)

Intervals	Mean residues 2,4D in water (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	NR	BDL	BDL	BDL	BDL
24 HAS	NR	BDL	BDL	BDL	BDL
48 HAS	1.234	BDL	BDL	BDL	BDL
54 HAS	1.097	0.223	BDL	BDL	BDL
72 HAS	0.027	0.206	BDL	BDL	BDL
4 DAS	0.010	0.328	0.02	BDL	BDL
5 DAS	BDL	BDL	0.04	BDL	BDL
7 DAS	BDL	BDL	BDL	BDL	BDL

HAS-Hours after spraying, DAS- Days after spraying, BDL- Below detectable limit paddy field and field outlet while the residues in drainage channel, stream and river were below detectable limit. The mean residue of 2,4-D were 0.883 and 1.25 ppm at 54 HAS while the residues in drainage channel, stream and river were below detectable limit. At 72 HAS the mean residue were 0.328 and 0.825 ppm respectively. The residues were below detectable limit in drainage channel, stream and river. When observed at 4 DAS, the mean residues of 2,4-D were 0.052 and 0.502 ppm in field and field outlet respectively. In the drainage channel residues of 2-4-D were detected at 0.02 ppm level. However, the residues were below detectable limit in stream and river. At 5 DAS the mean residues of 2,4-D were 0.017 ppm in paddy field while in field outlet, drainage channel, stream and river the residues were below the detectable level of 0.01ppm. At 7 DAS no detectable residue was present in samples of paddy field, field outlet, drainage channel, stream or river. Based on the residue decay curve, the half life of 2,4-D in soil was worked to be 0.59 days under Kuttanadu rice ecosystem.

4.5.1.2 Dissipation of 2,4-D in water

The mean residues of 2,4-D in water in rice field ecosystem are presented in Table 44. As dewatering is done before 2,4-D application and letting in water is done after 24 h, residues in water from the rice field was done at 48 HAS. At 48 HAS, the mean residues in water sample collected from the paddy field was 1.23 ppm. While in field outlet, drainage channel, stream and river the residues were BDL of

0.01 ppm. At 54 HAS the mean residues of 2,4-D was 1.097 ppm while in field outlet it was 0.223 ppm. After 72 h the mean residue was 0.027 ppm in water sample collected from paddy field while it was 0.206 ppm in field outlet.At 4 DAS, the mean level of 2,4 D in the rice field was 0.01 ppm while in the outlet was 0.328 ppm. In the drainage channel 2,4-D residue was detected at 0.02 ppm level. On 5 DAS, traces of 2,4-D residues were detected in rice field and field outlet while 2,4-D residues at 0.04 ppm level was observed in drainage channel at 5 DAS.

4.5.1.3 Dissipation of chlorpyriphos in soil

Table 45 represents the mean residues of chlorpyriphos in soil in rice ecosystem of Kuttanadu when observed at different intervals. The inititial deposit (2 HAS) of chlorpyriphos in soil in paddy field was recorded to be 0.561 ppm when analysed at two hours after spraying. In the field outlet ,drainage channel, stream and river the residues of chlorpyriphos were below detectable limit of 0.005 ppm. When observed at 24 HAS, the mean residues of chlorpyriphos in soil of paddy field was 0.389 ppm while that in drainage channel was 0.165 ppm. In the drainage channel, stream and river the residues of chlorpyriphos were below detectable limit. At 48 HAS the mean residues of chlorpyriphos was observed to be 0.28 ppm. In field outlet and drainage channel the mean residues of chlorpyriphos were recorded to be 0.122 and 0.05 ppm respectively. In the stream and river the residues of chlorpyriphos were below detectable limit. The mean

Table 45. Residues of chlorpyriphos in soil at intervals in Kuttanadu rice ecosystem
(Field situation-1)

Period	Mean residues chlorpyriphos in soil (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	0.561	BDL	BDL	BDL	BDL
24 HAS	0.389	0.165	BDL	BDL	BDL
48 HAS	0.281	0.122	0.05	BDL	BDL
72 HAS	0.151	0.102	0.02	BDL	BDL
4 DAS	0.057	BDL	BDL	BDL	BDL
5 DAS	0.037	BDL	BDL	BDL	BDL
7 DAS	0.025	BDL	BDL	BDL	BDL
10 DAS	0.016	BDL	BDL	BDL	BDL
15 DAS	BDL	BDL	BDL	BDL	BDL
30 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying, BDL- Below detectable limit

Table 46. Residues of chlorpyriphos in water at intervals in Kuttanadu rice ecosystem
(Field situation-1)

Period	Mean residues chlorpyriphos in water (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	NR	BDL	BDL	BDL	BDL
24 HAS	0.320	BDL	BDL	BDL	BDL
48 HAS	0.240	0.916	0.01	BDL	BDL
72 HAS	0.210	0.89	BDL	BDL	BDL
4 DAS	0.652	0.02	BDL	BDL	BDL
5 DAS	0.005	0.001	BDL	BDL	BDL
7 DAS	BDL	BDL	BDL	BDL	BDL
10 DAS	BDL	BDL	BDL	BDL	BDL
15 DAS	BDL	BDL	BDL	BDL	BDL
30 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying, BDL- Below

detectable limit

residues of chlorpyriphos at 72 HAS were recorded as 0.151 ppm. In the field outlet chlorpyriphos residues (0.102ppm) could be detected while in drainage channel residues of chlorpyriphos at 0.02 ppm level was observed. In the stream and river the residues of chlorpyriphos were below detectable limit When analysed at 4 DAS the mean residues of chlorpyriphos in soil of paddy field was observed to be 0.057 ppm while in field outlet the residues were BDL. In the drainage channel, stream and river the residues of chlorpyriphos were below detectable limit. At 5 DAS chlorpyriphos residue were 0.037 ppm while in field out let only traces of chlorpyriphos were observed. On 7, 10, and 15 DAS chlorpyriphos residues were detected in soils of rice field alone and the values were 0.025, 0.016 and 0.006 ppm respectively. On 30 DAS residues of chlorpyriphos were below detectable level in all the components analysed.

4.5.1.4 Dissipation of chlorpyriphos in water

The dissipation of chlorpyriphos in water in rice ecosystem of Kuttanadu are presented in Table 46. The mean residues of chlorpyriphos in water in paddy field when recorded 48 HAS was 0.240 ppm. In the field outlet the mean residues were recorded to be 0.916 ppm. In the drainage channel, stream and river the residues of chlorpyriphos were below detectable limit of 0.001 ppm. At 72 HAS the mean residue of chlorpyriphos in water in paddy field been recorded as 0.210 ppm while that in drainage channel as 0.89 ppm. At 4

DAS the mean residues of chlorpyriphos was found to be 0.652 ppm while that in field outlet was 0.02 ppm. At 5 DAS, the mean the residues of chlorpyriphos was recorded to be 0.005 ppm in field and 0.001 in field outlet respectively. Beyond 5 DAS, the residues were BDL in all the samples analyzed.

4.5.1.5 Dissipation of acephate in soil and water of paddy ecosystem

The initial residues of acephate when recorded at two HAS was found to be 0.06 ppm in rice field of Kuttanadu (Table 47). No residue of acephate could be detected in field outlet, drainage channel, stream and river in Kuttanadu. When observed at 24 HAS, the residues were found to be 0.019 ppm. At 48 HAS, the mean residues were 0.01 ppm and there after the mean residues were below detectable level of 0.01ppm. Residue of acephate was below the detectable limit of 0.01 ppm in water samples collected from paddy field in all the occasions under study

4.5.1.6 Dissipation of hexaconazole in soil

The data on the dissipation of hexaconazole in paddy soil are presented in Table 48. The initial deposit (2 HAS) of hexaconazole in soil in paddy field was recorded to be 0.359 ppm when analysed at 2 HAS. In the field outlet only traces of hexaconazole residues was detected. In the drainage channel, stream and river the residues of hexaconazole were below detectable limit of 0.05 ppm. When observed

Table 47. Residues of acephate in soil and water at intervals in Kuttanadu rice ecosystem

(Field situation-1)

Intervals	Mean residues of acephate	
	soil	water
2 HAS	0.0595	NR
24 HAS	0.019	BDL
48 HAS	0.010	BDL
72 HAS	BDL	BDL
4 DAS	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying NR :Not recorded

BDL- Below detectable limit

Table 48. Residues of hexaconazole in soil at intervals in Kuttanadu rice ecosystem (Field situation-1)

Intervals	Mean residues hexaconazole in soil (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	0.359	BDL	BDL	BDL	BDL
24 HAS	0.307	0.13	0.10	BDL	BDL
48 HAS	0.162	0.11	BDL	BDL	BDL
72 HAS	0.106	BDL	BDL	BDL	BDL
4 DAS	BDL	BDL	BDL	BDL	BDL
5 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying, BDL- Below detectable limit

at 24 HAS , the mean residues of hexaconazole in soil of paddy field was 0.307 ppm while that in field out let and drainage channel were 0.13 and 0.10 ppm respectively. In the soil samples collected from stream and river, the residues of hexaconazole were below detectable limit. At 48 HAS, the mean residues of hexaconazole in paddy field and drainage channel was 0.162 ppm and 0.11 ppm respectively. The residues of hexaconazole were below detectable limit in soil samples from stream and river.

4.5.1.7 Dissipation of hexaconazole in water

The mean residues of hexaconazole in water in paddy field when recorded at 24 and 48 HAS were 0.250 and 0.209 ppm (Table 49). In the field outlet the mean residues were recorded to be 0.01 ppm. In the drainage channel, stream and river the residues of chlorpyriphos were below detectable limit of 0.01 ppm. At 72 HAS residue of hexaconazole in water field was 0.145 ppm while in field out let, drainage channel, stream and river were below detectable level and there after that the residues were BDL in all the water samples analysed.

4.5.1.8 Dissipation of Lambda cyhalothrin in rice plant

Lambda cyhalothrin residues were not detected in any of the soil or water samples collected from the rice fields of Kuttanadu. The data indicated that the initial deposit (at 2 HAS) in rice plant was 4.097 ppm (Table

Table 49. Residues of hexaconazole in water at intervals in Kuttanadu rice ecosystem (Field situation-1)

Intervals	Mean residues of hexaconazole (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	NR	NR	BDL	BDL	BDL
24 HAS	0.250	NR	BDL	BDL	BDL
48 HAS	0.209	0.01	BDL	BDL	BDL
72 HAS	0.145	BDL	BDL	BDL	BDL
4 HAS	BDL	BDL	BDL	BDL	BDL
5 DAS	BDL	BDL	BDL	BDL	BDL
7 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying NR :Not recorded

BDL- Below detectable limit

Table 50. Residues of lambdacyhalothrin in soil, water and rice plant at intervals in Kuttanadu rice ecosystem (Field situation-1)

Intervals	Mean residues in ppm		
	Soil	Water	Plant
2 HAS	BDL	BDL	4.097
24 HAS	BDL	BDL	3.402
48 HAS	BDL	BDL	2.269
72 DAS	BDL	BDL	1.871
5 DAS	BDL	BDL	0.923
7 DAS	BDL	BDL	0.488
10 DAS	BDL	BDL	0.031
15 DAS	BDL	BDL	BDL
30 DAS	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying, BDL- Below detectable limit

50). At 24 HAS the mean residues of lambdacyhalothrin in rice plants were detected to be 3.402 ppm At 48 HAS, the mean residues of lambda cyhalothrin was 2.269 in rice plant. At 72 HAS, the mean residues of lambda cyhalothrin in rice plant was 1.871 ppm. At 5 DAS the mean residues of lambda cyhalothrin was 0.923 ppm in. At 7 DAS and 10 DAS, the residues of lambda cyhalothrin were 0.488 and 0.031 respectively. Beyond 10 days the residues were below detectable limit of 0.02ppm.

4.5.2 Field situation II

The results of dissipation of the residues of 2,4-D, monocrotophos, triazophos and methyl parathion both in the soil and water following application in the rice field to the adjoining field outlet, drainage channel, stream and finally to the river are presented in tables 51 to 56.

4.5.2.1 Dissipation of 2,4-D in rice soils of Kuttanadu : Field situation II

Mean residues of 2,4-D in soil are presented in Table 51. The data indicated that the initial deposit (at2 HAS) in soil in paddy field was 3.583 ppm while it was 2.13 ppm in the field outlet. Residues were below the detectable level of 0.01 ppm in the drainage channel. Samples from stream and river did not show the presence of 2,4-D residues. At 24 HAS, the mean residues were 2.851 ppm and 1.88 ppm respectively paddy field and field outlet respectively. The samples

Table 51. Residues of 2,4-D in soil at intervals in Kuttanadu rice ecosystem

(Field situation-II)

Intervals	Paddy field (ppm)	Field outlet	Drainage channel	Stream	River
2 HAS	3.583	2.13	BDL	BDL	BDL
24 HAS	2.851	1.88	BDL	BDL	BDL
48 HAS	2.385	1.02	BDL	BDL	BDL
72 HAS	1.948	0.99	BDL	BDL	BDL
78 HAS	0.746	0.521	BDL	BDL	BDL
4 DAS	0.026	BDL	BDL	BDL	BDL
5 DAS	0.011	BDL	BDL	BDL	BDL
7 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying, BDL- Below detectable limit

Table 52. Residues of 2,4-D in water at intervals in Kuttanadu rice ecosystem

(Field situation-II)

Intervals	Mean residues 2,4D in water (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 hr	NR	BDL	BDL	BDL	BDL
24 hr	NR	BDL	BDL	BDL	BDL
48 hr	1.22	BDL	BDL	BDL	BDL
72 hr	0.992	0.01	BDL	BDL	BDL
78 hr	0.01	BDL	BDL	BDL	BDL
4 Days	Traces	BDL	BDL	BDL	BDL
5 Days	Traces	BDL	BDL	BDL	BDL
7 Days	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying

BDL- Below detectable limit

collected from drainage channel, stream and river did not show the presence of the herbicide residues. At 48 HAS the mean residues of 2,4-D were 2.385 and 1.02 ppm respectively in paddy field and field outlet while the residues in drainage channel, stream and river were below detectable limit. The mean residue of 2,4-D were 1.948 and 0.99 ppm at 72 HAS while the residues in drainage channel, stream and river were below detectable limit. At 78 HAS the mean residues were 0.746 and 0.521 ppm respectively. The residues were below detectable limit in drainage channel, stream and river. When observed at 4 DAS the mean residues of 2,4-D were 0.026ppm in the rice field. In the field outlet, drainage channel, river and stream residues of 2-4-D were below detectable limit. At 5 DAS the mean residues of 2,4-D was 0.011 ppm in paddy field while in field outlet, drainage channel, stream and river the residues were BDL. At 7 DAS the residues of 2,4-D were BDL in paddy field, field outlet, drainage channel, stream and river. 162

4.5.2.2 Dissipation of 2,4-D in water in rice ecosystem

The mean residues of 2,4-D in water are presented in Table 52. At 48 HAS, the mean residues in water sample collected from paddy field was 1.22 ppm. In filed outlet traces of 2,4-D residues were observed. While in drainage channel, stream and river the residues were BDL of 0.01 ppm. At 72 HAS the mean residues of 2,4-D was 0.99 ppm while in field outlet the residues at the level of 0.01 ppm was observed. After 78 h, the mean residues were 0.01ppm in water sample collected from paddy field. In the drainage channel 2,4-D residues were below detectable level. On 4 DAS, traces of 2,4-D residues were detected in rice field and while that in field outlet, drainage channel, stream and river the residue were below below detectable limit.

4.5.2.3 Dissipation of monocrotophos in soil and water of paddy ecosystem

The initial residues of monocrotophos when recorded at two hours after spraying was found to be 0.297 ppm in soil in rice field of Kuttanadu (Table 53). No residue of monocrotophos could be detected in field outlet, drainage channel, stream and river in Kuttanadu. When observed at 24 HAS the residues were found to be 0.290 ppm. At 48 hours after spraying the mean residues were 0.162 ppm in soil in rice field of Kuttand. At 72 HAS mean residues of monocrotophos was 0.059 ppm. Beyond 72 hours the residue of monocrotophos was below the detectable limit of 0.01 ppm in soil samples collected from paddy field, field outlet, drainage channel, stream and river. Water samples when collected at 48 HAS the residue of monocrotophos was 0.038ppm. At 72 HAS the mean residues were 0.023 ppm. Beyond 72 hours the residues of monocrotophos was below detectable limit of 0.01 ppm in the water samples collected.

Table 53. Residues of monocrotophos in soil and water at intervals in Kuttanadu rice ecosystem (Field situation-II)

Period	Mean residues in	
	Soil (ppm)	water (ppm)
2 HAS	0.297	NR
24 HAS	0.290	0.042
48 HAS	0.162	0.038
72 HAS	0.059	0.023
4 DAS	BDL	BDL
5 DAS	BDL	BDL
7 DAS	BDL	BDL
10 DAS	BDL	BDL
15 DAS	BDL	BDL
30 DAS	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying

NR :Not recorded, BDL- Below detectable limit.

Table 54. Residues of triazophos in soil at intervals in Kuttanadu rice ecosystem
(Field situation-II)

Period	Mean residues triazophos in soil (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	0.388	BDL	BDL	BDL	BDL
24 HAS	0.324	0.151	BDL	BDL	BDL
48 HAS	0.204	0.08	BDL	BDL	BDL
72 HAS	0.122	BDL	BDL	BDL	BDL
5 DAS	0.100	BDL	BDL	BDL	BDL
7 DAS	0.063	BDL	BDL	BDL	BDL
10 DAS	0.032	BDL	BDL	BDL	BDL
15 DAS	BDL	BDL	BDL	BDL	BDL
30 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS : Days after spraying, BDL- Below detectable limit

4.5.2.4 Dissipation of triazophos in soil of paddy ecosystem

Table 54 presents the mean residues of triazophos in soil in paddy ecosystem of Kuttanadu when observed at different intervals. The initial deposit (2 HAS) of triazophos in soil in paddy field were recorded to be 0.388 ppm when analysed at 2 HAS. In the field outlet only traces of triazophos was detected. In the drainage channel, stream and river the residues of triazophos were below detectable limit of 0.01 ppm. When observed at 24 HAS, the mean residues of triazophos in soil of paddy field was 0.324 ppm while that in drainage channel was 0.151 ppm. In the drainage channel, stream and river the residues of triazophos were below detectable limit. At 48 HAS the mean residues of triazophos was observed to be 0.204 ppm. In field outlet the mean residues of triazophos were recorded to be 0.08 ppm. In drainage channel, the stream and river the residues of chlorpyriphos were below detectable limit. The mean residues of triazophos at 72 HAS were recorded as 0.122 ppm. In the field outlet only traces of triazophos residues could be detected. In the drainage channel stream and river the residues of triazophos were below detectable limit. At 5 DAS triazophos residues were observed to be at the level of 0.1 ppm while in field out let only traces of triazophos were observed. At 7 DAS and there after residues of triazophos was detected only in paddy soil of the field and in the field outlet, drainage channel, stream and river the residues were below detectable limit. The mean residue of triazophos in soil of paddy field was recorded to be 0.063 ppm. The residues were reduced to the tune of 0.032 ppm in the paddy fields when observed at 10 DAS.On 30 DAS no residues of chlorpyriphos could be detected in any of the components analysed.

4.5.2.5 Dissipation of triazophos in water of paddy ecosystem

The dissipation of triazophos in water in rice ecosystem of Kuttanadu are presented in Table 55.The mean residues of triazophos in water in paddy field when recorded 48 hours after spraying was 0.230 ppm. In the field outlet the mean residues were recorded to be 0.05 ppm. In the drainage channel, stream and river the residues of triazophos were below detectable limit of 0.01 ppm. At 72 HAS, the mean residue of triazophos in water (table 56) in paddy field been recorded as 0.12 ppm while that in drainage channel as 0.02 ppm.. At 4 DAS, the mean the residues of triazophos was recorded to be 0.01 ppm in field while the residues were below detectable limit in field outlet, drainage channel, stream and river. Beyond 5 DAS the residues were BDL in all the samples analyzed.

4.5.2.6 Dissipation of Methyl parathion in rice plant

Residues of methyl parathion were not detected in any of the soil or water samples collected from the rice fields of Kuttanadu in the dissipation study. The data indicated that the initial deposit (at 2 HAS) of methyl parathion in rice plant was 2.131 ppm (Table 56**).** At 24 hours after

Table 55 Residues of triazophos in water at intervals in Kuttanadu rice ecosystem
(Field situation-II)

Period	Mean residues triazophos in water (ppm)				
	Rice field	Field outlet	Drainage channel	Stream	River
2 HAS	NR	BDL	BDL	BDL	BDL
24 HAS	0.446	BDL	BDL	BDL	BDL
48 HAS	0.230	0.05	BDL	BDL	BDL
72 HAS	0.12	0.02	BDL	BDL	BDL
4 DAS	0.01	BDL	BDL	BDL	BDL
5 DAS	BDL	BDL	BDL	BDL	BDL
7 DAS	BDL	BDL	BDL	BDL	BDL
10 DAS	BDL	BDL	BDL	BDL	BDL
15 DAS	BDL	BDL	BDL	BDL	BDL
30 DAS	BDL	BDL	BDL	BDL	BDL

HAS: Hours after spraying, DAS :Days after spraying, NR :Not recorded,
BDL- Below detectable limit

Table 56. Residues of methyl parathion in soil ,water and rice plant at intervals in Kuttanadu rice ecosystem (Field situation-II)

Intervals	Residues in ppm		
	Soil	Water	Plant
2 HAS	BDL	BDL	2.131
24 HAS	BDL	BDL	2.015
48 HAS	BDL	BDL	1.201
72 DAS	BDL	BDL	0.869
5 DAS	BDL	BDL	0.451
7 DAS	BDL	BDL	0.205
10 DAS	BDL	BDL	0.011

HAS: Hours after spraying, DAS :Days after spraying, BDL- Below detectable limit

spraying (HAS) the mean residues of methyl parathion in rice plants were detected to be 2.015 ppm. At 48 HAS, the mean residues of methyl parathion was 1.201 in rice plant. At 72 HAS, the mean residues of methyl parathion in rice plant was 0.869 ppm. At 5 DAS, the mean residues of methyl parathion were 0.451 ppm. At 7 DAS and 10 DAS the residues of methyl parathion were 0.205 and 0.01 respectively. Beyond 10 days the residues were below detectable limit of 0.01 ppm.

4.6 Estimation of dermal exposure of spraymen to pesticides during application of pesticides in Kuttanadu rice ecosystem

The data on the dermal exposure of one fungicide, hexaconazole and one insecticide, triazophos to the spraymen engaged on the operations at the tillering stage of paddy crop were studied (Table 57). The mean deposit of hexaconazole on the uncovered portions of body of the spray man during pesticide application was recorded to be 2.14 $\pm$ 0.109 mg $m^{-2}h^{-1}$ while that of triazophos was 3.55$\pm$ 0.131 . Utilizing the value for mg $m^{-2}h^{-1}$, the total dermal exposure of pesticide per man was calculated to be 3.76$\pm$0.116 and 6.20$\pm$0.152 for hexaconazole and triazophos respectively. From this data the average dermal exposure per kg body weight per day was calculated taking in to account the average weight of sprayman as 62 kg and average hours of spraying as six. Thus the average dermal exposrue per kg per day was worked out to be 0.36$\pm$0.011 and 0.59 $\pm$0.015 for hexaconazole and triazophos respectively.

Table 57. Dermal exposure of spray men to pesticides during application in Kuttanadu rice ecosystem

Pesticide	Dose	Dermal exposure		
		mg man^{-2} hour^{-1}	mg man^{-2} hour^{-1}	mg kg^{-1}day^{-1}
Hexaconazole	0.05	2.14 $\pm$ 0.109	3.76$\pm$0.116	0.36$\pm$0.011
Triazophos	0.05	3.55$\pm$ 0.131	6.20$\pm$0.152	0.59$\pm$0.015

DISCUSSION

5. DISCUSSION

Kuttanadu, the rice bowl of Kerala is distinctive in its geographical features with unique low lands 1-2.5 meters below mean sea level (MSL). Nearly 55000 ha of wetlands in Kuttanadu are available for paddy cultivation year-round. The poor drainage conditions prevailing in the region make it unsuitable for other crops. The land use pattern in Kuttanadu is depicted in Figure 4. Rice cultivation in Kuttanadu is of an intensive nature compared to any other part of the state. The main crop season of Kuttanadu is the 'Puncha' which extends from October/November to March/April. During this period water is drained out of the rice fields using indigenous pump 'Petti and Para' and paddy is grown under submerged conditions. Majority of the farmers grow high-yielding varieties of rice necessitating the use of high levels of chemical inputs. Due to the high temperature and relative humidity prevailing, the area is prone to infestation by many pests which demand regular application of pesticides in the region. Outbreaks of different pests have been reported from Kuttanadu at times by different workers (Ambikadevi *et al.*, 1998; Nalinakumari *et al.*, 2002). The excess use of agricultural inputs including pesticides in Kuttanadu was reported by Alexander and Krishnakumari (1990).

Pesticide use in Kuttanadu had always been suspected of being a major contributor to environmental pollution. The usual practice of draining the field to drainage channels, streams and rivers may cause river and lake contamination. Persistent pesticides may remain in the plants and

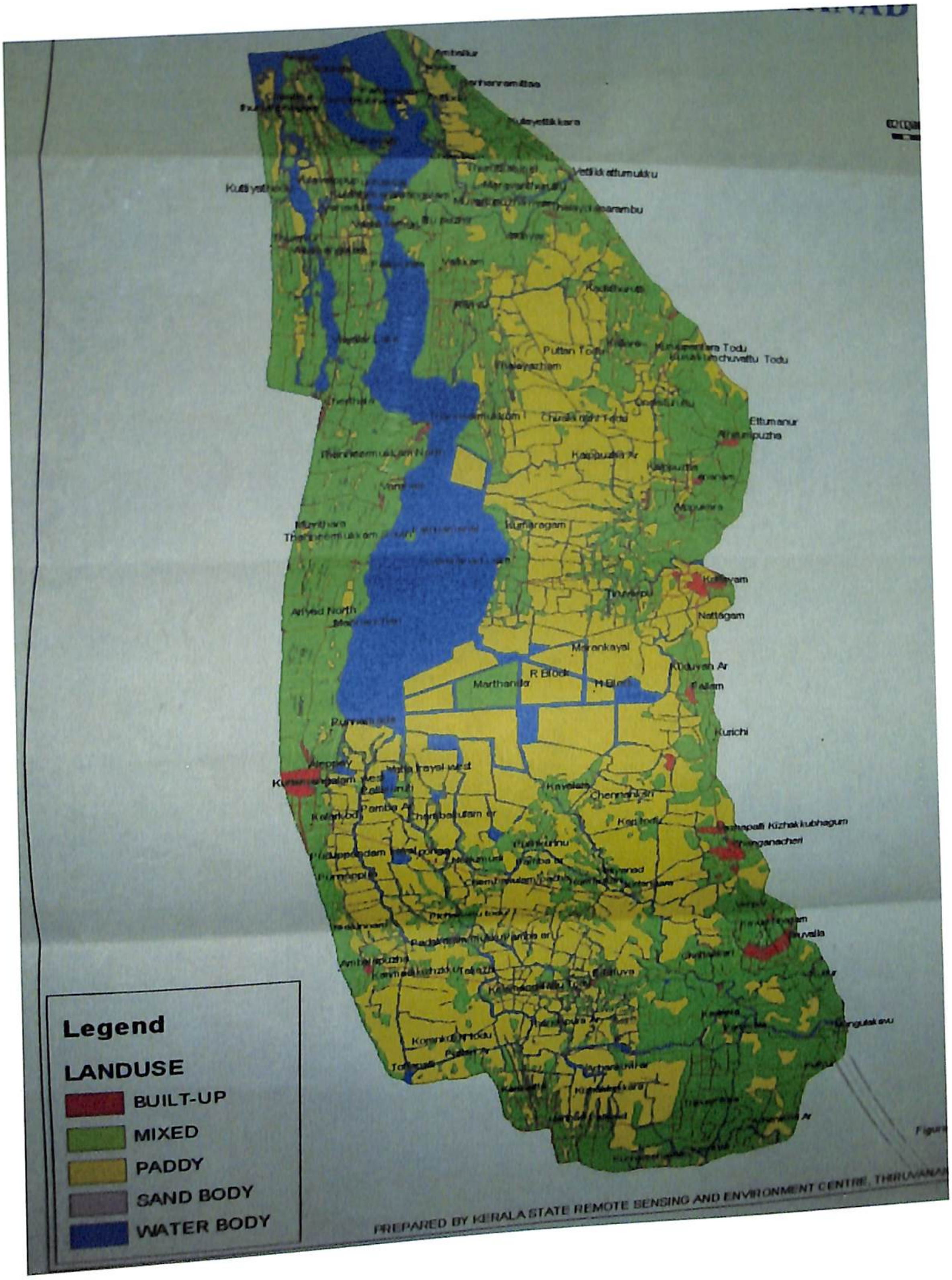


Figure 4. Land use pattern of Kuttanadu

contaminate food and feed from the agro system. Further, the use of toxic pesticides pose potent threat to farmer health in the region.

Recent studies conducted by Devi, (2009) revealed that farmers often spray hazardous pesticides at higher doses than recommended causing high risk to farmer health and environment.

Earlier Dinham (1993) reported frequent cases of cancer of the lip, stomach, skin and brain, lymphoma, leukemia and multiple myloma from the Kuttanadu linking the same to high pesticide use in the area. Rakhesh (1999) studied the externalities associated with pesticides in Kuttanadu and found that pesticide poisoning leads to both explicit and implicit costs for the applicator/ farmer. Recently Devi (2009) reported skin problems as the major hazard linked to pesticide use in the region. Further, eye irritation, vision and breathing problems, dehydration, vomiting, cramps and diarrhea are reported by her from among the farm workers.

The present study was undertaken with a view to assess the environmental as well as the health impacts of pesticides in the rice ecosystem in Kuttanadu. Extensive and intensive monitoring studies were conducted to review the on-paddy and off-paddy effects of pesticides in rice ecosystem.

Extensive studies included the survey on pesticide use and its impact on farmer health and monitoring of paddy soils, water, rice grains, and non-target components like fish, milk, duck and animal meat in Kuttanadu. Intensive studies included dissipation pattern of pesticides in rice soil and water in different components of Kuttanadu rice ecosystem and the effect on pest and natural enemy complex. Studies were also undertaken to assess the dermal exposure of pesticides during the application to the spray men in Kuttanadu so as to highlight the risk involved and to persuade them to follow Good Agricultural Practices related to the use of pesticides.

5.1 SURVEY

A detailed survey was carried out in the major rice growing tracts of Kuttanadu in the catchments of river Pampa, Manimala and Meenachil to study the pesticide use pattern and pesticide consumption prevalent in the region during 2007-09 and the same was compared with those prevailed during 2002-03. The salient findings are discussed below.

The pesticide use pattern (Table 1) followed by the farmers in Kuttanadu revealed that among the 23 popular pesticides being used in Kuttanadu, 18.18 per cent are herbicides, another 18.18 per cent are fungicides and 65.22 per cent are insecticides. When the pesticides were classified according to their toxicity, 8.70 per cent belonged to the group 'less toxic' marked as green, 39.13 per cent to the group 'moderately toxic' marked as blue, another 39.13 per cent to the group 'highly toxic' marked as yellow and 17.39 per cent to 'extremely toxic' marked as red. Survey also revealed that the most popular herbicide, insecticide and fungicide were 2, 4-D, acephate and hexaconazole respectively. Similar studies conducted by Devi (2010), revealed a similar trend in pesticide use pattern in Kuttanadu during the period

2004-05. She reported the use of the organochlorine insecticide, lindane in Kuttanadu during 2004-05. However, in the present study it was observed that the use of organochlorines have been completely given up in Kuttanadu. A decline in consumption of organochlorines was observed all round India. Mullen *et al.,* (1997) observed that the pesticide risk to environment is related to the amount and type of active ingredient, its toxicity, mobility and persistence characteristics. If farmers reduce the total quantity of pesticide active ingredient applied, but simultaneously substitute highly twice toxic mobile and persistent chemicals in relatively lower quantities, it is equally unsafe to the environment. Majority of insecticides currently being used in Kuttanadu viz., acephate (73.33 %), monocrotophos (57.5 %), chlorpyriphos (45.83 %), quinalphos (43.33 %), dimethoate (27.5 %), phorate (26.67 %), methyl parathion (24.17 %), are organophosphates and belong to either highly toxic or extremely toxic group. Majority of the chemicals being used in India belong to organo phosphate group which can cause health damages to human beings. In the present study it was observed that restricted use pesticides (RUP) like monocrotophos and methyl parathion were popular and available in many regions in Kuttanadu. Monocrotophos is an organo phosphorous systemic insecticide extremely toxic to birds and poisonous to mammals. All applications of this chemical were discontinued in the US since 1998 (Devi, 2010). In India, this chemical is banned for use in vegetables. Devi, (2010) again reported that the consumption of methyl parathion was increased by 16.83 per cent in Kerala during the period 1991-92 to 2007-08.

The frequency of herbicide application by farmers of Kuttanadu during the year 2002-03, 2007-08, and 2008-09 is represented in Figure 5. The data revealed that 4.17 and 15 per cent respectively of the farmers were not using any herbicide during 2007-08 and 2008-09 while 61.67 and 62.5 per cent of farmers adopted one time application during the same period. Farmers who applied herbicide twice during crop season during 2007-08 and 2008-09 were 33.33 and 19.17 per cent respectively. In the year 2007-08, three times application of herbicide was adopted by 0.83 per cent of farmers while none of the farmers resorted to more than two applications during 2008-09. When the data is compared with those prevailed during 2002-03, it is observed that 49.17 per cent each of the farmers resorted to either no application or one time application while 1.6 per cent of farmers applied herbicides two times during the crop season and no farmer applied herbicides more than twice. The mean number of applications of herbicides in different locations of the catchments in Kuttanadu ranged from 0.7 to 1.8 during 2007-08 and 0.5 to1.5 in 2008-09 while the corresponding value ranged from 0.2 to 1.0 during 2002-03.

The present study revealed the fact that the herbicide use and frequency of application were increasing over the years as evidenced by the increase in number of applications during 2007-08 and 2008-09

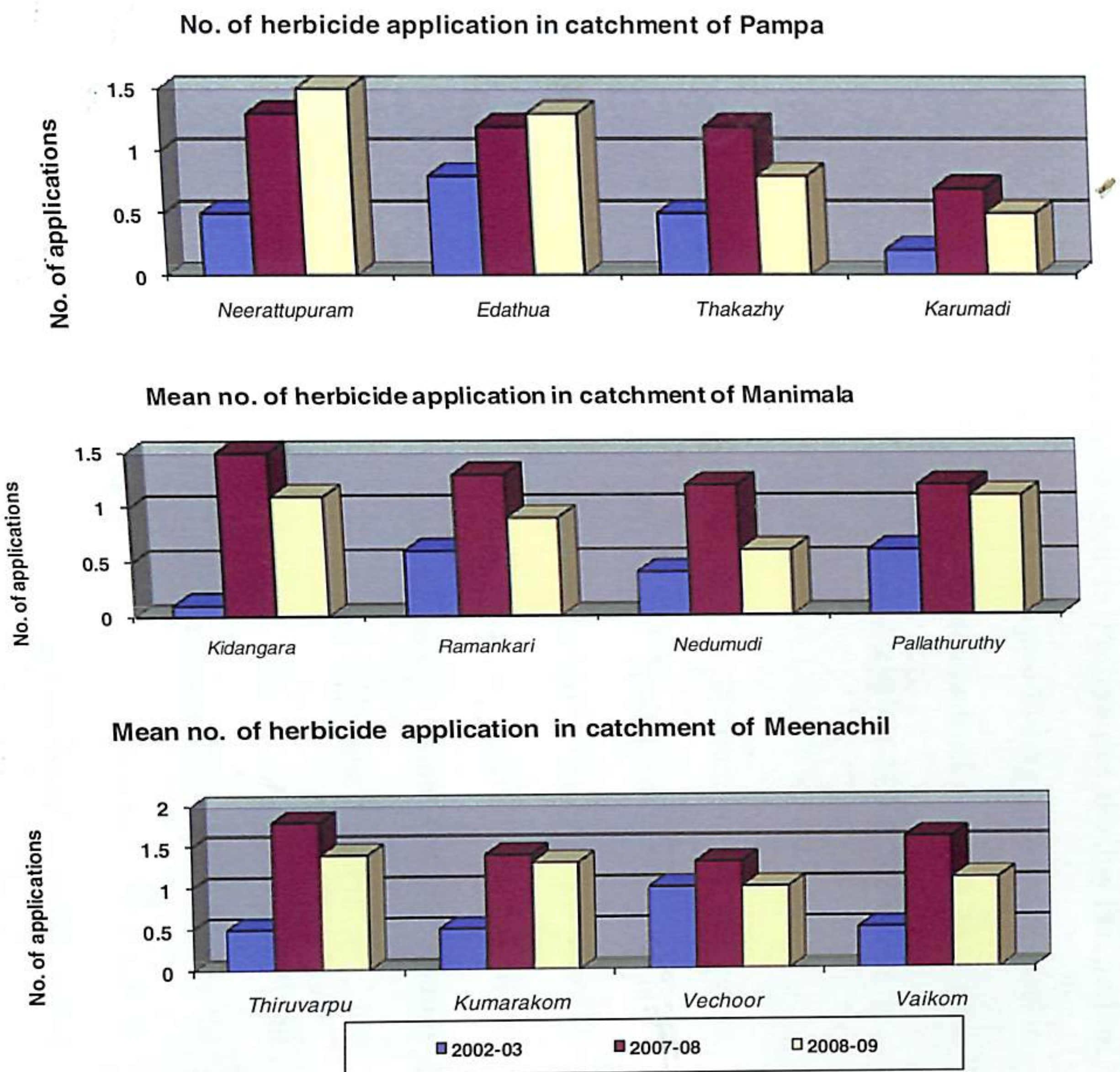


Figure 5. Number of herbicide application in different years

compared to that existed during 2002-03. The main reasons for increasing herbicide usage in Kuttanadu are scarcity and high cost of labour prevalent in Kerala. Similar trend was noticed in many other rice growing countries. Moody, 1990 reported that the use of herbicide was increasing rapidly all over the world due to the escalating cost of labour. However, a study conducted in Philippines during the period from 1966 to 1990, it was observed that the average herbicide use in rice growing provinces in Philippines had remained constant over time with an average of one application per season. (Warburton *et al.*, 1993).

The present study revealed that in Kuttanadu the selective herbicide 2, 4- D, which destroys broad leaved weeds, is the most popular and extensively used herbicide. Introduction of target specific herbicides like cyhalofop-methyl, which exclusively destroys *Echinochloa* had contributed to the increase in number of applications of herbicides in the region. The feedback from the farmers revealed that proper water management practices play a major role in weed control in the region. Herbicide application becomes inevitable in regions where proper water management practices cannot be adopted. Further, increasing land conversion cause serious threat to proper water management practices which indirectly influence the weed management and there by herbicide use in Kuttanadu.

When the insecticide use pattern among the rice farmers in Kuttanadu was studied during the year 2007-08, it was observed that 35.83 per cent of farmers adopted one time application of insecticides while 21.67 per cent farmers applied insecticides twice, 10.00 per cent applied thrice and 3.33 per cent applied more than thrice (Figure 6). No insecticide was used by 29.17 per cent of farmers. During the year 2008-09, among the 120 farmers 40.00 per cent each of the farmers adopted one time and two time application of insecticides during the crop season under study. Ten per cent farmers were not using any insecticide at all in their field. Insecticide application twice and thrice was done by 3.33 and 6.67 per cent of the farmers respectively. In 2002-03 among the 120 farmers 32.50 per cent adopted one time application of insecticide while 37.50 per cent farmers applied insecticides twice during the crop season. Insecticide was not applied by 7.50 per cent of farmers. Insecticide application thrice and more than thrice was done by 16.60 and 5.80 per cent of the farmers respectively. When the insecticide consumption over the years was compared it could be observed that unlike the situation prevailed during 2002-03, none of the farmers attempt more than thrice application of insecticides in 2007-08 and 2008-09. The present trend in reduction in insecticide application may be explained in terms of the popularization of the high yielding rice variety Uma (MO-16) with high yield potential and moderate resistance to stem borer and BPH

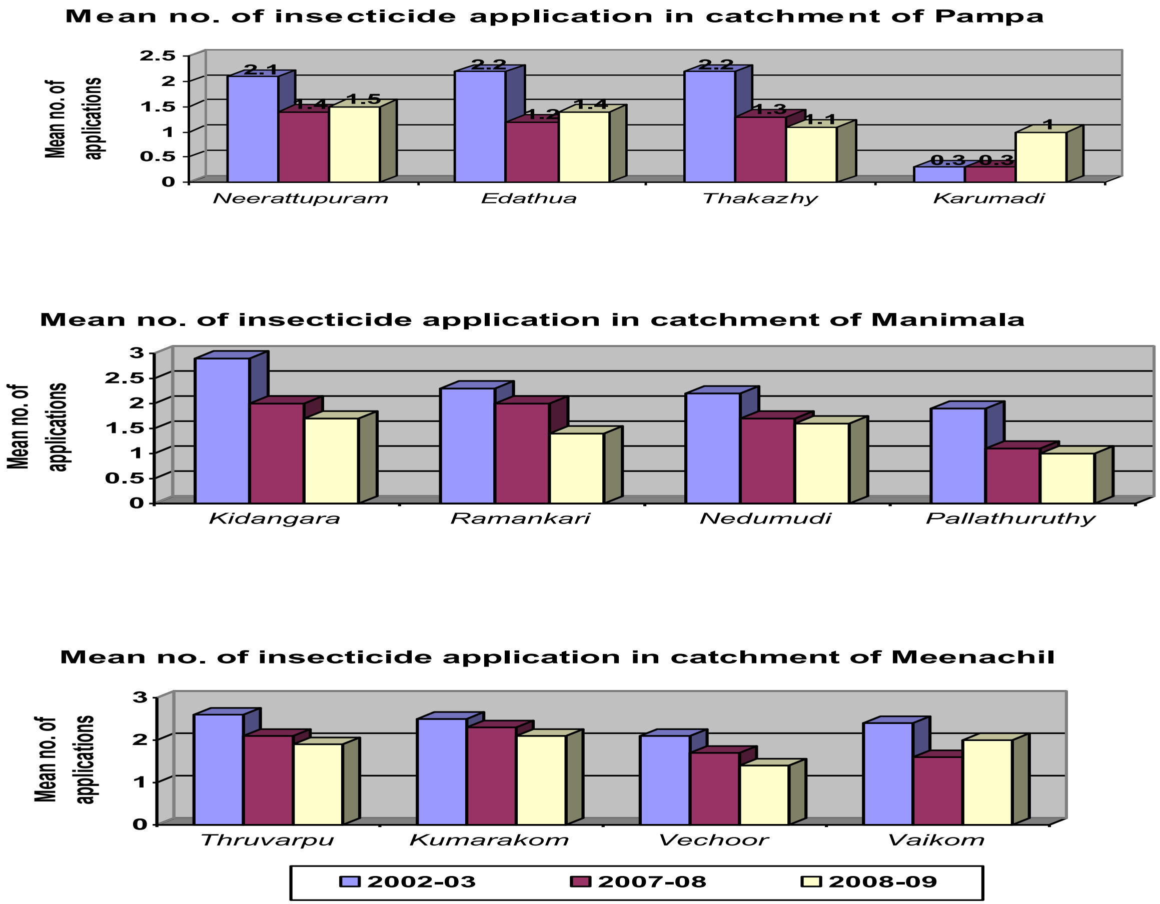


Figure 6. Number of insecticide application in different yerar

supplemented by the shift towards integrated pest managementpractices. The most popular photo insensitive rice variety Uma released from Rice Research Station, Moncompu has high yield attributes like plant height, more number of productive tillers, tolerance to pest like stem borer, brown plant hopper and gall midge (Devika *et al*.,2004). The diminishing trend of insecticide application frequency observed in the present study matches with the trend reported by Warburton *et al*. (1993) who reported increasing awareness of pest threshold and host plant resistance among farming community in Philippines.

When the fungicide use pattern in the region was examined, the data revealed that in 2007-08 majority (68.33 %) of the farmers limited fungicide application once in the crop season. Among the farmers surveyed 23 (19.17 %) farmers were not using any fungicide at all while 12.50 per cent were applying fungicides twice per crop season and none of the farmers resort to more than two applications (Figure 7). During 2008-09, it was observed that 63.33 per cent of the farmers limited fungicide application once in the crop season. Among the 120 farmers in the catchments, 29.00 (24.17 %) farmers were not using any fungicides at all while 15.00 (12.50) per cent were applying fungicides twice during the crop season. When fungicide application was compared with that existed during 2002-03 it was seen that 51.60 per cent of farmers limited the application once in a crop season. Among 120 farmers 35

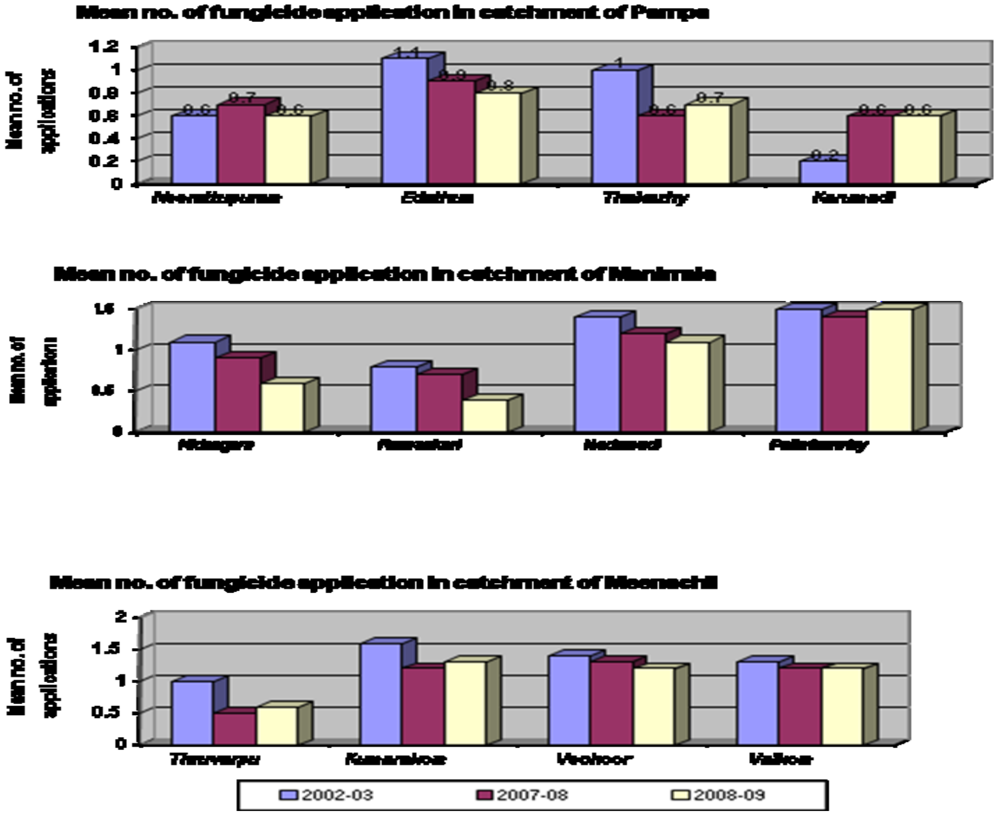


Figure 7. Number of fungicide application in different years

(19.10 %) farmers were not using any fungicides at all while 29.10 per cent farmers were applying fungicides twice during one crop season. The frequency of fungicide application did not change over the years under the study. During 2002-03, 2007-08 and 2008-09 majority of farmers applied fungicides once in a crop season. There were no reports on the incidence of any disease epidemic over these years in the region which substantiates the present finding of the fungicide consumption in the region. The frequency of fungicide application by farmers of Kuttanadu during the year 2007-08, and 2008-09 in comparison to 2002-03 is also represented in Figure 6.

Considering the various sources of knowledge of pesticides for the farmers, 45.83 per cent depend on pesticide sales men for information regarding pesticide use. Only 31.67 per cent farmers depend on government personnel for their knowledge regarding pesticide use. The feed back from the farmers revealed that the main reason why they depend on pesticide sales men is that they only have up to date information about the availability of pesticides in the depot for the immediate use by the farmers. Non availability of Govt. subsidy for purchase of plant protection chemicals also contribute towards the dependence of the farmers to the personnel in the pesticide depot. A more or less similar situation where farmers depend on company representative/pesticide sales men for obtaining technical information regarding crop protection was reported by Shetty (2004) from the states of Karnataka and Andhra Pradesh. Forty per cent of the paddy farmers from the districts of Bellary and Raichur in Karnataka and 41.00 per cent of cotton farmers from Guntur and Warangal districts of Andhra Pradesh depend on company representative/pesticide sales men. Warburton *et al*. (1993) also observed the dependence of rice farmers in Philippines towards government personnel and pesticide sales men than pesticide labels for their knowledge about pesticide application rate. The dependence of farmers towards company representatives/ pesticide sales men shows the weak performance or absence of efficient extension machinery in the region for the timely support and guidance. However, the picture in different catchments are examined in detail it could be seen that in the catchment of Manimala, majority of the farmers (45%) depend on Government machinery which indicates the effective functioning of such establishments in the region.

The insecticide application is initiated by the farmers (54.17 %) when mere presence of pest is observed in the field. Actually the farmers are not aware of the economic threshold concept and are not ready to follow that concept. For example, farmers resort to prophylactic soil application of cartap hydrochloride granules against rice stem borer irrespective of their occurrence.

The concept of IPM was known to majority of farmers surveyed. However, the extent of adoption was low. Though 70.00 per cent of farmers are aware of IPM, only 45.00 per cent adopted the practice either partial or in total. This finding was in contradiction to that reported by Shetty *et al.* (2008) form Punjab where 34.00 per cent of the farmers surveyed were unaware of IPM practices and only less than 5.00 per cent of them had been following complete IPM measures. The level of adoption of IPM in Kuttanadu rice ecosystem observed in the present study was much higher than the overall national average of 2.00 per cent as reported by Shetty *et al.,* (2008). This may be due to the high literacy rate among the farmers and the impact of IPM training programmes conducted in the region. According to the farmers the extent of adoption of IPM can be further increased by ensuring the availability of quality products in sufficient quantities at the required time and by reducing the price of inputs. Though the concept of IPM is well conceived by the farmers, the concept of Good Agricultural Practices was not known to 92.50 per cent of farmers.

The extent of adoption of protective measures by the spray men in the Kuttanadu rice ecosystem during the different stages of implementation of plant protection measures was collected during the survey (Figure 8). Guidelines for safe pesticide application recommend the use of protective gadgets which include face mask with replaceable filters, goggles, head cover, rubber gloves, full sleeved shirts and full pants and boots. None of the spray men in the region follow the guidelines in toto. In a study conducted in the same region by Devi during 2004-05 also depicted a similar situation. Previous report from

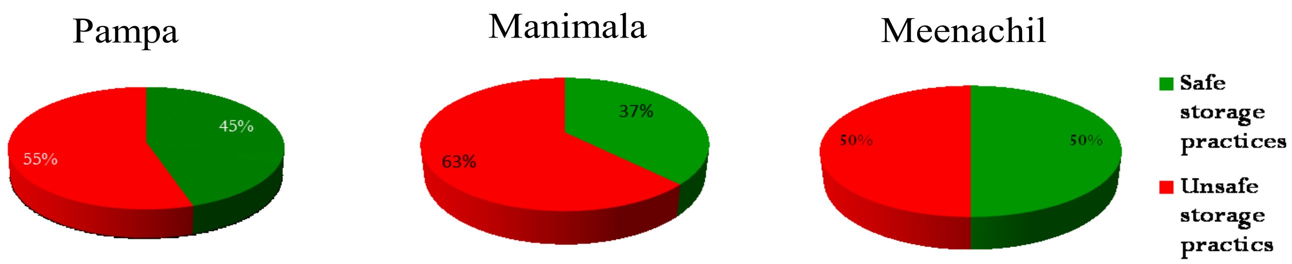
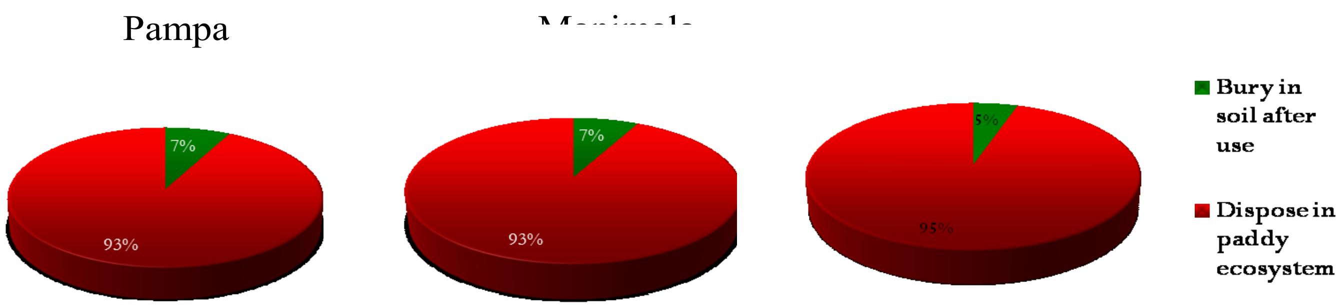


Figure 8. Extent of adoption of pesticide storage practices in catchments



other parts of the country also revealed that about 69.00 per cent of the spray men covered their face with towel or cloth particularly to avoid the smell and did not use any other protective clothing while spraying pesticides (Shetty, 2004). Earlier reports from other countries are also in agreement with the present situation prevailed in the region. Jayarethnam *et al.* (1987) and Sivayoganathan *et al.* (1995) from Sri Lanka and Yassin *et al.* (2002) from Palestine reported similar situations. Further, the findings of some other studies conducted in the developing countries also supported this aspect (Wilson, 1998; Gomes *et al*., 1999, Murphy *et al*.,1999; Salameh *et al.*, 2004 and Atreya, 2007). In the present study, it is observed that 30.00 per cent of the spray men are not at all using any of the protective measures during handling of plant protection chemical though rest of them follow different measures which give protection at varying levels. The protective measures adopted by the farmers include covering the nose with hand towels instead of face mask (66.67 %) and use of footwear instead of boots (3.33 %). Among the forty spray men, 56.57 per cent reported that non-availability of protective clothing was the major reason for not adopting safety measures during pesticide application. They were also of opinion that the protective clothing decreases their speed of operation and efficiency in spraying. Devi (2009) identified the cost factor as the major reason for not using protective gadgets. The spray men become reluctant to adopt the recommended gadgets and opt for cheaper substitutes. Further general lethargy and the discomfort associated with their use under the hot and humid conditions and under the puddled paddy land conditions were also identified as the reasons for non-adoption of proper protective gadgets. International Code of Conduct on the Distribution and Use of Pesticides give specific guidelines for effective utilization of toxic pesticides. According to article 3.5 of the International Code of Conduct, pesticides whose handling and application require the use of comfortable and expensive protective clothing and equipment should be avoided, especially in tropical climates for use by small - scale users. However, this article is not enforced in almost all the tropical countries including India. No monitoring mechanisms are in force to ensure pesticide handling operations as per GAP.

Pesticide labels are important means of information regarding the dose, timing of application, quantity, waiting period, antidote etc. Among the pesticide spray men, 65.00 per cent reported that the labels are too small to be read. Only 16.00 per cent of pesticide operators are reading the label information before the start of spraying. In a country like India with multiple languages and dialects, label norms and directions are important. Labels should be in local languages and include pictographs that clearly identify product hazards, appropriate modes of storage and handling and other precautions that users should be aware of. Further, the International Code of Conduct on Reducing

Health Hazards, article 5.2.2.5 states the importance of clear and concise labeling and article 11.1.17 states that advertisements should encourage purchasers and users to read the label carefully or have the label read to them if they cannot read.

In addition to the pesticide contamination due to direct spraying, the wet land ecosystem is further contaminated by the unscientific practices followed by the farmers. In the study it was observed that more than 92 per cent of the respondents were disposing the pesticide container unscientifically (Figure 10). They were leaving the containers in the paddy ecosystem itself (Plate 4). More than 50.00 per cent of the farmers were draining off the wash water to the paddy field or irrigation channel which leads to wastage of pesticides and aggravate the risk for environment. The present finding on unscientific disposal of the empty pesticide containers in to paddy ecosystem and draining of wash water to the paddy field warrant an urgent necessity for farmer training on proper pesticide handling, storage and disposal which could minimize unnecessary exposure to the chemicals. Improper disposal of pesticide containers by farmers including the reuse of the containers for storing kerosene, diesel etc from different parts of the country are reported by Shetty (2004).

Direct health aspects of the sixty spray men were also collected during the survey and the data presented in Figure 11. Headache and dizziness were the main problem for majority (43.3%) of the

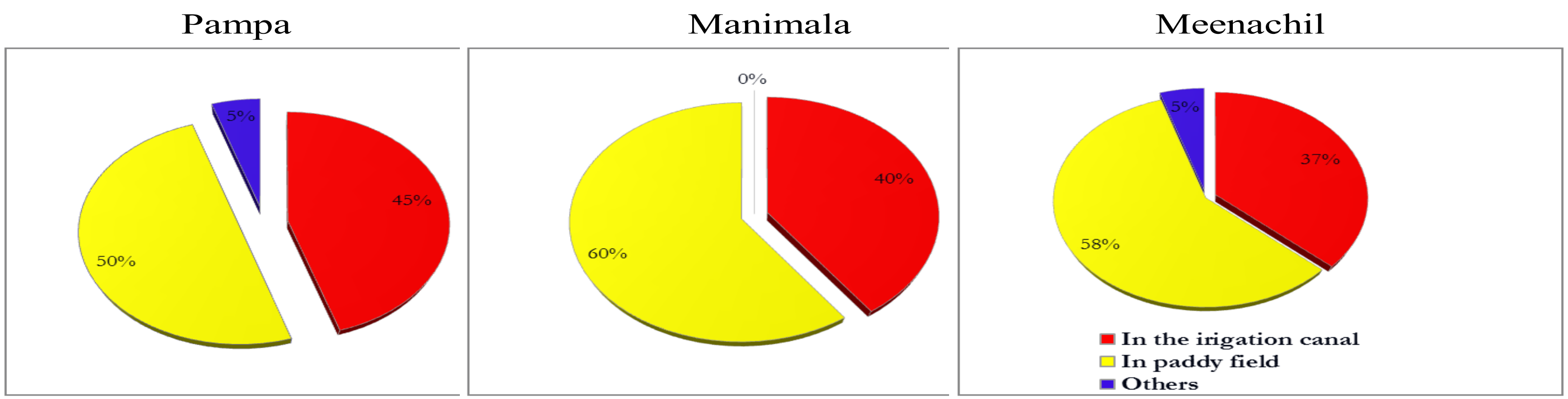


Figure 10. Disposal of pesticide wash water after spraying in catchments

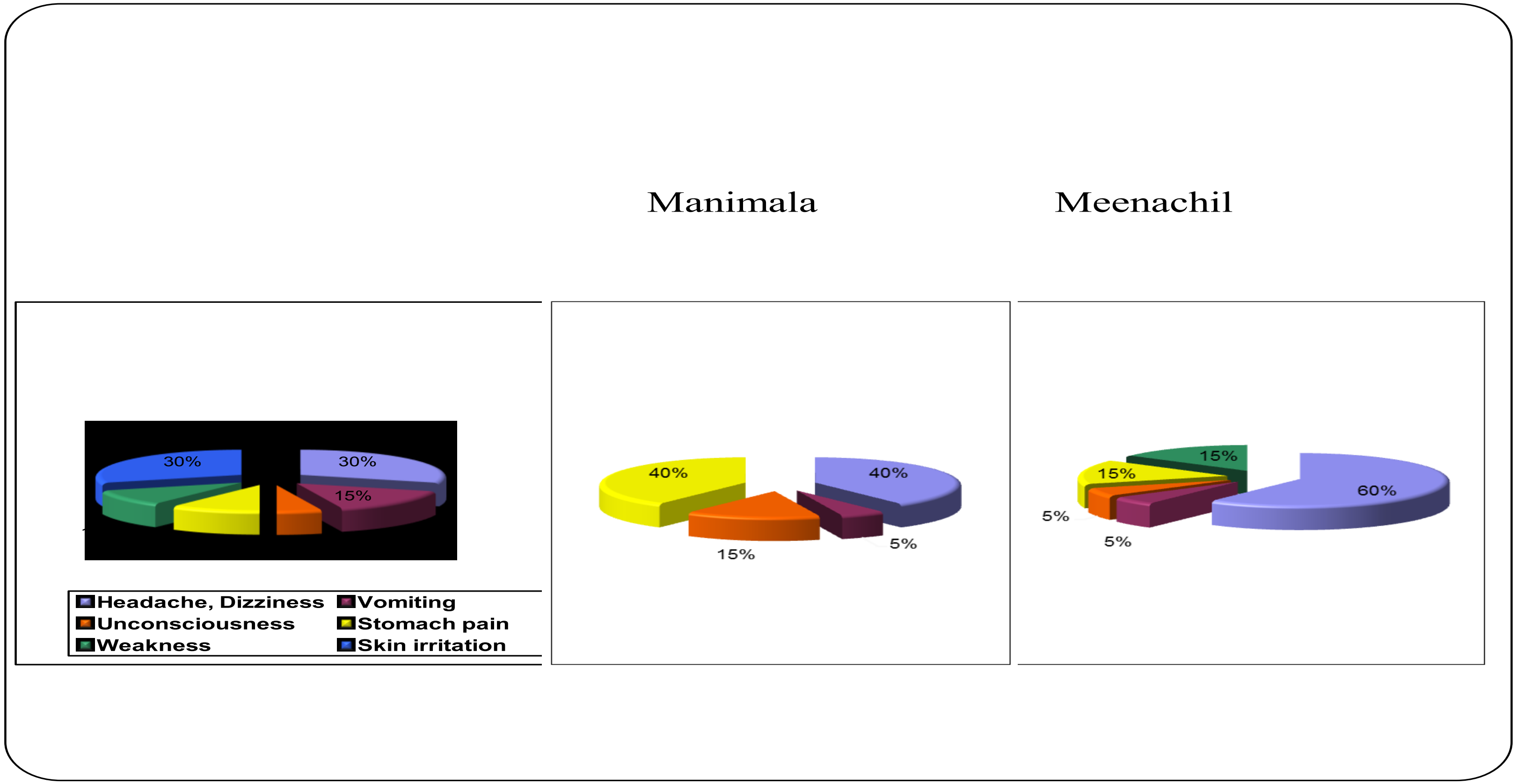


Figure 11. Health problems as reported by spray men in different catchments

Plate 5. Pesticide packets / bottles disposed in paddy eco-system

respondents. Dermal disorders on exposure to pesticides were another important health hazard. Among the sixty farmers 28.33 per cent were suffering from dermal diseases. Another health problem faced by the pesticide spray men was stomach pain (10.00%) and general weakness (8.30 %). During long periods of pesticide application unconsciousness occurred in case of 3.33 per cent of spray men. Similar studies conducted by Krishna (2001) in Kuttanadu revealed that majority of farmers (60%) were reported to be suffering from health problems caused by pesticides. He reported that among the health hazards induced by pesticides, the skin allergy and headache were most prominent in Kuttanadu. Devi (2009) also reported that skin problems, eye irritation and vision problems, symptoms like nausea, giddiness, breathing problems, dehydration, vomiting, cramps, convulsions and diarrhea occurred among the spray men from the region.

Validation of multi residue methods

Developing sensitive and reliable analytical methods incorporating validation parameters such as repeatability, reproducibility and quality parameters such as precision, linearity and detection limits capable of detecting pesticides at very low levels is an urgent requirement in chemical analysis especially in monitoring of pesticide residues in food and environmental samples to produce accurate and reproducible results (Garg *et al.,* 2009). By means of validation procedure the performance of the test method is investigated systematically for the production of accurate and reproducible results. In Kuttanadu rice ecosystem an array of pesticides which include herbicides, insecticides and fungicide are being used in the same crop frequently resulting in the possibility for multiple residues in the biotic and abiotic components of the ecosystem. Hence selection and validation of a suitable multi residue method to estimate the residues of commonly used pesticides is an urgent thrust. Therefore validation of multi residue methods for estimation of pesticide residue in water, soil, rice grains and paddy straw, fish and duck meat were performed. The quality parameters for method validation such as repeatability, reproducibility and quality parameters such as precision, linearity and detection limits for eleven pesticides prevalently used in Kuttanadu essential to assess the method were proved and are presented in Tables 15 to 30.

Three internationally accepted multi residue methods for pesticide residue analysis in water were tested during the study. Among the three methods tested (Table 14) the micro separator method was found to be the simplest and cheapest which involves minimum quantity of organic solvents and chemicals. A single drawback observed in this case was that this method was not suited for estimation of residues of herbicides like 2,4-D which is more polar and acidic in nature. Therefore micro separator method was not considered for further studies.

The method which was found to be more suited in the present study was the one developed by Hernandez *et al.,* (1993) with slight modification. The repeatability in terms of recovery percentage of the method was determined at four levels, 100 ppb, 50 ppb, 10 ppb and 1 ppb. The reproducibility in terms of recovery percentage of the method done at three consecutive days was determined at three levels, 100 ppb, 50 ppb and 10 ppb. The limit of quantification was fixed as 1ppb for phorate, lindane, methyl parathion, malathion, chlorpyriphos and alpha endosulfan while it was fixed as 10 ppb for ediphenphos, lambda cyhalothrin, deltamethrin, dimethoate and hexaconazole. For 2,4-D the LOD was fixed as 10ppb.

In case of paddy soil, new multi residue methods for pesticide residue analysis in soil – Acetone extraction method was developed. The method was compared with Soxhlet extraction method. Between the two methods, the acetone extraction method was found to be more accurate with high recovery and precision. In Soxhlet extraction method, pre-drying of soil is essential before extraction. This may increase the adsorption of pesticide molecules to the clay colloids. They reported that organic herbicides when applied were adsorbed to the soil colloids of highly acidic clay soil than in neutral soils. Tejada *et al.*, (1990) reported that adsorption of pesticide molecules like chlorpyriphos to soil colloids was up to 91 per cent suggesting strong affinity to soil. The low recovery from soil following soxhlet extraction method (54.96 -78.06%) observed in the present study may be explained in terms of the clayey nature of soil of Kuttanadu which is highly acidic and rich in organic matter. Hence a new method using acetone extraction method which uses acetone as solvent was developed and validated. This method gave recoveries ranging from 65 to 108 per cent with RSD <20 %. This method can be adopted for multi residue analysis of paddy soils in Kuttanadu.

The multi residue methods for paddy grain and straw were also validated. The repeatability and reproducibility of all the 11 pesticides were established with acceptable recovery and RSD <20 %. The repeatability in terms of recovery percentage of the method developed for paddy grains was determined at three levels, 0.2.0.1 and 0.05 ppm, the per cent recovery being 56.75 to 92.95, 63.69 to 94.89 and 55.55 to 98.70 respectively. Similarly, the repeatability in terms of recovery percentage of the method developed for straw was determined at three levels, 0.2.0.1 and 0.05 ppm, the per cent recovery being 59.87 to 94.72, 65.89 to 95.35 and 59.12 to 99.73 respectively.

The multi residue methods for fish was validated for eleven candidate pesticides with mean recovery per cent of 66.33 to 99.74 per cent at 0.2 ppm fortification level. The corresponding recovery level at 0.10 and 0.05ppm were 62.76 to 99.98 and 59.44 to 93.18 respectively. In a similar study the recovery of MRM of fish was observed as 85.00 to 91.00 per cent for organo chlorines at 0.5 ppm and 1ppm. The results of MRM for meat revealed that the method was efficient with mean recovery of 62 to 112 per cent. Recently in Japan a more sensitive multi residue method was developed and validated for 185 pesticides and the LOQ was set as 0.01 ppm.

5.3 Monitoring of pesticide residues in biotic and abiotic components of Kuttanadu rice eco-system

Farmers in Kuttanadu quite often resort to chemical control measures irrespective of recommendations or guidelines. The dosage, time of application and type of chemical used in the region are much higher and varied. When pesticides are applied on rice plants, many droplets fall between the foliage especially in the inter row spaces and contaminate the irrigation water and soil (Mathew, 1982). Over 50 per cent of pesticides applied to a crop reach to soil during the applications for plant protection leading to the pollution of soil and water bodies (Aswathi *et al.,*2002). When water is drained off from the fields to the canals the pesticide molecules along with water may reach adjacent canals, streams, rivers and finally the lakes. In Kuttanadu frequent draining of water from rice field may lead to the discharge of pesticide molecules to nearby channels and from there to stream and then to river or reservoir resulting in non-point source of contamination. The persistence of toxic pesticide residues in soil and water adversely affect soil heath, aquatic life and quality of water (Kumar *et al.,* 1995). Persistent pesticides may further remain in the plants and contaminate food and feed from the agro system. In this context extensive monitoring studies were conducted in Kuttanadu to explore the extent of contamination of biotic and abiotic factors in the paddy eco-system of Kuttanadu. The results of the monitoring studies are presented in Table 33-38. The mean residues if any, present in soil and water in different non-point sources of contamination viz., paddy field, field out let, drainage channel, stream and the final destination river were analyzed during the different growth stages of rice. The food and feed from the rice – ecosystem in Kuttanadu were also analyzed. In the study, residues of herbicides and insecticides could be detected in water and soil during the early stages of growth period *viz*., seedling stage and tillering stage in the rice field, field out let and drainage channel.

Out of the 30 samples of water collected from the catchment of Pampa during the seedling stage, residues of herbicide 2, 4-D could be detected in eight samples at the level of 0.01 to 0.054 ppm. Among the eight samples, residues were detected in three samples each from paddy field and field outlet and two from drainage channel. During the tillering stage, out of the 30 samples, residues of monocrotophos could be detected in three samples with a range of 0.01 to 0.03 ppm in the paddy field and field outlet. No residue could be detected in water samples collected during the booting stage and milky stage at catchment of Pampa.

Out of the 30 samples collected from the catchment of Manimala during the seedling stage, residues of the herbicide 2,4-D could be detected in seven samples at the level of 0.02 to 0.056 ppm. Among the seven samples, residues were detected in four samples from paddy field, two from field outlet and one from drainage channel. During the tillering stage, out of the 30 samples, chlorpyriphos (0.005 to 0.02 ppm) could be detected in one sample each in paddy field, field outlet and drainage channel. A similar trend was seen in the case of triazophos residues (0.01 to 0.03 ppm). During the booting stage residues of malathion could be detected in two samples to the level of 0.05 to 0.1 ppm. No residue could be detected in water samples collected during the milky stage at catchment of Manimala.

Out of the 30 samples of water collected from the catchment of Meenachil during the seedling stage, residues of herbicide 2, 4-D could be detected in seven samples at the level of 0.01 to 0.035 ppm. Among the seven samples, residues were detected in four samples in the paddy field, two in field outlet and one in drainage channel. During the tillering stage, out of the 30 samples, monocrotophos (0.01 ppm) was detected in one sample from the field out let and phorate residues (0.038 to 0.062 ppm) could be detected in two samples in the paddy field and field outlet. No residue could be detected in water samples collected during the booting stage and milky stage at catchment of Meenachil.

The examination of the data on the transport of the residues in water and soil from the point of application to the different destinations *viz*. field outlet, drainage channel, stream and finally to river at different stages of crop growth are examined it could be seen that during the seedling stage, residues of 2,4 -D was present in 12 water samples collected from paddy field, seven samples from field out let and four samples from drainage channel. Samples from stream and river were totally free from residues. In the tillering stage, five samples each from paddy field and field outlet and two samples from drainage channel showed the presence of chlorpyriphos and triazophos residues. As in the previous case, samples from stream and river were totally free from residues. At booting stage one sample each from rice field and field outlet showed residues of malathion while samples from drainage channel, stream and river were free from residues. On the contrary, at milky stage no reside was seen in water samples either at point of application or at different destinations.

The examination of the data on the transport of the residues in soil from the point of application to the different destinations *viz.,* field outlet, drainage channel, stream and finally to river at different stages of crop growth are examined it could be seen that during the seedling stage, residues of 2, 4 -D was present in six soil samples collected from paddy field and five samples from field out let. Samples from drainage channel, stream and river were totally free from residues. In the tillering stage, five samples from paddy field and two samples each from field outlet and drainage channel showed the presence of chlorpyriphos and triazophos residues. Samples from stream and river were totally free from residues. At booting stage and milky stage none of the samples from rice field, field outlet, drainage channel, stream and river showed the presence of residues.

The pesticides that are carried with the effluence of the paddy field as surface run off or adsorbed on suspended solids would end up first in drainage channel. It is at this point when the pesticides begin to move out of their target area and travel towards larger non target bodies like drainage channel, stream and river. The concentration of pesticides become diluted and reach below detectable level as it move from the paddy field to destinations like drainage channel, stream and river. It seems that some degree of degradation or dilution with water caused the decrease in pesticide concentration as the pesticides moved from the point of application to the different destinations. The absence of residues in stream and river in the present study reveal that not much threat exist in the region as far as the major water bodies are concerned. However, the present findings on the presence of 2,4-D residues in field outlet and drainage channel demand a further extension of water holding period of herbicides from the current practice of 48-72 h as a good agricultural practice for controlling the herbicide runoff from paddy fields .

Similar studies had been conducted and reported from several parts of the world. Monitoring studies for pesticide concentrations in river systems in Japan detected several herbicides commonly used in paddy fields (Nakamura,1993). They conducted seasonal and spatial studies on pesticide residues in surface waters of the Shinano river in Japan. Among the total of 53 chemicals found, 22 were herbicides, 15 were insecticides, 11 were fungicides and 5 were metabolites. Water samples from irrigation tanks and drinking water source from major paddy growing areas and areas of intensive pesticides use in Sri Lanka and found that out of 544 samples analyzed eight were positive for either chlorpyriphos (0.22 to 0.542 ppm) or diazinon (0.012-0.15 ppm) and one sample contained dimethoate (0.014 ppm). On the contrary to the above findings, pesticide residues were not detected in any of the river systems in Kuttanadu in the present study. The data revealed that herbicides are the major contaminants during the initial stages of growth while insecticide residues are detected during the succeeding stages. Anasco *et al*. (2010) measured pesticide residues in five freshwater areas that are directly affected by rice paddy effluents in southern Japan to determine their maximum concentrations and temporal variations which showed that herbicides had relatively higher concentrations in the earlier stages of the rice planting season, while insecticides and fungicides had relatively higher concentrations at the later stages. Table 56 and 57 presents the mean residue of herbicides

Table 58 Pesticide residues in samples of water in Kuttanad rice eco-**system**

Sampling details	Catchment of			
	Pampa	**Manimala**	**Meenachil**	**Total**
Seedling stage				
No. of samples analyzed	30	30	30	90
Detected	8	8	7	23
Pesticides detected (ppm)	2,4-D 0.01 to 0.054	2,4-D 0.01 to 0.056	2,4-D 0.01 to 0.05	
Tillering				
No. of samples analyzed	30	30	30	90
Detected	3	6	3	12
Pesticides detected (ppm)	Monocrotophos (0.01 to 0.030)	Chlorpyriphos (0.005 to 0.02) Triazophos (0.01 to 0.03)	Monocrotophos (0.01) Phorate (0.062 to 0.038)	
Booting				
No. of samples analyzed	30	30	30	90
Detected	Nil	2	Nil	2
Pesticides detected (ppm)		Malathion (0.05 to 0.1)		
Milky stage				
No. of samples analyzed	30	30	30	90
Detected	Nil	Nil	Nil	

Table 59 Pesticide residues in samples of soil in Kuttanad rice eco-system

Sampling details	Catchment of			
	Pampa	Manimala	Meenachil	**Total**
Seedling stage				
No. of samples analyzed	30	30	30	90
Detected	7	8	4	19
Pesticides detected (ppm)	2,4-D (0.018 to 0.2) Dimethoate (0.3)	2,4-D (0.01 to 0.056)	Dimethoate (0.01 to 0.03) 2,4-D (0.01 to 0.02)	
Tillering				
No. of samples analyzed	30	30	30	90
Detected	nil	7	2	9
Pesticides detected (ppm)		Chlorpyriphos (0.1 to0.32), Triazophos (0.2 to0.32) Quinalphos (0.05)	Carbaryl (0.32) Phorate sulphone (0.502)	
Booting				
No. of samples analyzed	30	30	30	90
Detected	nil	nil	nil	nil
Pesticides detected				
Mean residues in ppm (range)				
Milky stage				
No. of samples analyzed	30	30	30	90
Detected	nil	nil	nil	nil

and insecticides in water and soil during the early stages of growth period viz., seedling stage and tillering stage in the rice field, field out let and drainage channel.

Out of the 30 samples of soil collected from the catchment of Pampa during the seedling stage, residues of the herbicide 2, 4-D and the insecticide dimethoate could be detected in six samples at the level of 0.18 to 0.2 ppm and 0.3 ppm respectively. Among the six samples residues were detected in three from paddy field, two from the field outlet and one from the drainage channel. During the tillering stage, out of the 30 samples, no residue could be detected in soil samples collected during the tillering, booting and milky stages at catchment of Pampa.

Out of the 30 samples collected from the catchment of Manimala during the seedling stage, residues of herbicide 2, 4-D could be detected in two samples at the level of 0.01 to 0.2 ppm. One sample each from paddy field and from field outlet showed the presence of residues. During the tillering stage, out of the 30 samples, chlorpyriphos (0.01 to 0.32 ppm), triazophos (0.02 to 0.32ppm) and quinalphos (0.05 ppm) could be detected in seven samples in paddy field, field outlet and drainage channel. No residue could be detected in soil samples collected during the booting and milky stage at catchment of Manimala.

Out of the 30 samples collected from the catchment of

Meenachil during the seedling stage, residues of the herbicide 2, 4-D and the insecticide, dimethoate could be detected in four samples at the level of 0.01 to 0.03 ppm and 0.01 to 0.02 ppm respectively. During the tillering stage, out of the 30 samples, carbaryl (0.32 ppm) and phorate sulphone (0.50 ppm) could be detected in two samples in the paddy field. No residue could be detected in soil samples collected during the booting stage and milky stage at catchment of Meenachil.

The overall data on the residues in soil revealed the predominance of the herbicide 2,4-D over other pesticides in Kuttanadu. The frequent occurrence of 2,4-D in soil samples may be attributed to the fact that organic herbicides when applied were adsorbed more by highly acid clay soil than neutral soils.

When the data on residues of pesticides in biotic components were examined it was found that none of the samples of paddy grains and straw showed the presence of any residue which may be due to the lengthy interval between application and harvest and due to rapid degradation of pesticides in tropical low land environment of the ecosystem. Pingali (1995) from Philippines reported similar results and found that in flooded rice small amounts of pesticides applied at recommended rates and intervals do not persist beyond the crop growing period. Monitoring of residues in the non target components of the ecosystem like fish, egg, duck meat and beef also indicated the absence of residues in all the components. However, Tejada *et al*, 1995 reported the presence of chlorpyriphos residues in heart and muscle of duck fed in rice ecosystem of Philippines. Samanta (2006) reported DDT and its metabolites, HCH, heptachlor, endosufan and aldrin in fish samples collected from River Ganga in West Bengal. However, in the present study residues of organochlorine pesticides are not at all detected in any of the components of the ecosystem.

5.4.4.4. Impact of conventional and IPM practices on major pests, natural enemies and neutrals

The concept of IPM refers to the conditions associated with the pest control such as environmental impact of indiscriminate use of plant protection chemicals, introduction of management concept of keeping the population of pests below economic threshold level and the overall ecological considerations. Impact of IPM on the population of pest and natural enemy complex has to be compared in terms of the one prevalent in a situation which follow conventional methods of pest control so as to advocate its importance. The population of pests in IPM and conventional (Non IPM) plots in Kuttanadu are presented in Table 39. The data revealed that the mean population of the above pests observed at occasions of 25 DAS, 45 DAS and 65 DAS in IPM plots were higher than that of the conventional plots.

The population of natural enemies and neutrals in IPM and non-IPM plots in Kuttanadu at different intervals is presented in Table 40. The data revealed that the mean number of natural enemies *viz.*, *C.lividipennis, Agriocnemis,* dragon flies, *Tetragnatha, L.psuedoannulata, Tetrastichus, and Telenomus* in IPM plots were higher than that of non-IPM plots at 25 DAS, 45 DAS and 65 DAS.

The mean population of neutrals *viz* .,culicids, chironomids and house flies in IPM plots were higher than that of non-IPM plots at different intervals.

The pest to defender ratio (P/D ratio) was calculated to be 1.031 and 1.1406 at 25 DAS respectively in IPM and non-IPM plots. At 45 DAS the P/D ratio was 1.83 and 2.118 respectively for IPM and non-IPM plots. The P/D ratio was found to be 1.76 and 3.037 respectively for IPM and non-IPM plots. The mean per cent damage of rice stem–borer and rice leaf roller in IPM (Table 41) was higher than non-IPM plots. However, the mean grain and straw yield (Table 42) in IPM plots was observed to be higher than that of the non- IPM plots which may be due to the abundance of natural enemies in a pesticide free environment.

The data on the residues revealed that the mean level of residues in the paddy grains, straw, meat, fish and soil samples were below the detectable level of 0.01ppm when sampled at the time of harvest from both in the IPM and non-IPM plots. This is in agreement with the findings of Mukherjee and Arora (2011) who studied the impact analysis of IPM programmes in rice by estimation of pesticide residues. The residues in all the grain samples of paddy were below detectable limits.

5.5 Dissipation of pesticides in Kuttanadu rice ecosystem

The surface drainage/run off of paddy water containing appreciably high concentrations of pesticides is responsible for pollution of water bodies in rice ecosystem. The draining of pesticide residues from the paddy field to the river system through field outlet, drainage channel and stream was undertaken in the present study and the dynamics of commonly used pesticides in Kuttand rice ecosystem are discussed below. In the present study, dissipation of one herbicide (2-4-D), six insecticides *viz.* chlorpyriphos, acephate, monocrotophos, triazophos, lambdacyhalothrin, methyl parathion and one fungicide (hexaconazole) were studied.

In the first experiment the herbicide 2,4-D was applied in the field at 17 DAS and water was drained off the field before spraying. After 24 h water was let in and allowed to remain for up to 48 h. After 48 h the water was drained off the field.

Dissipation pattern of 2,4-D residues revealed that residues reached below the detectable level of 0.01 ppm on 7DAS in soil in the paddy field. The initial deposit of 3.602 ppm reached BDL on the seventh day with a half life of 0.59 days. The residues of 2,4-D in the field outlet was lower than those existed in the field, the initial deposit being 2.62 which reached BDL in 5 DAS. In the drainage channel 2,4-D residues were BDL except on the fourth day which recorded 0.02 ppm. The presence of residues on the fourth day may be explained in terms of the practice of draining the rice field on second day after 2,4-D application. Both in stream and river no residue was detected in all the occasions. The probable cause for the absence of residues may be due to the heavy discharge rate of water into these bodies where by the low level of residues present in rice field and outlet get diluted to non-detectable levels. The mean residues of 2,4-D in water were 1.234 ppm at 48 HAS and became BDL on 5 DAS. The 2,4-D residues were detected in the field outlet when observed at 54 HAS and got gradually declined to BDL on 4 DAS.

The half life observed in the present study is in agreement with the findings of Wilson *et al*. (1998) who reported a half life of less than one day in rice soils. The low half life values reported for paddy soil is explained in terms of moisture content of the soil which has a major effect on half life. The commonly used 2,4-D amine salts and 2,4-D esters are not persistent under most environmental conditions. Dissociation of 2,4-D amine salt is expected to be instantaneous under most environmental conditions and ester forms of 2,4-D transform and hydrolyze rapidly to the acid in natural soil and water in less than one day. Under normal conditions 2,4-D residues are not persistent in soil or water because of metabolism into compounds of non-toxicological significance and ultimately to forms of carbon.

In the second experiment, a slight change in water management practices was made. The herbicide 2,4-D was applied in the field at 17 DAS and water was drained off the field before spraying. After 24 h, water was let in and allowed to remain for up to 72 h. After 72 h, the water was drained off the field. In this situation, dissipation pattern of 2,4-D residues revealed that residues reached below the detectable level of 0.01 ppm on 7 DAS in the paddy field. The initial deposit of 3.583 ppm reached BDL on the seventh day with a half life of 0.57

days. The residues of 2,4-D in the field outlet was lower than those existed in the field, the initial deposit being 2.13 which reached BDL in 4 DAS. In the drainage channel 2,4-D residues were below at all intervals. Both in stream and river no residue was detected in all the occasions. The mean residues of 2,4-D in water were 1.22 ppm at 48 HAS and became BDL on 4 DAS. The 2,4-D residues were detected in field outlet when observed at 72 HAS and gradually declined to BDL on 3 DAS.

The data revealed that the quantity of pesticide residues present in the rice field, field outlet, drainage channel and river was influenced by water management practices prevalent in the field. In the present study a decrease in half life of 2,4-D was observed when the time of dewatering was extended. The rate of dissipation was faster and residues were low in field outlet and drainage channel.

The water management practices in the region are the key practice for controlling the pesticide discharge from paddy fields. In California, the water holding period requirement after pesticide application has successfully reduced the concentrations of rice pesticides in streams (Newharat, 2002). The effect of water management practices on pesticide behavior in paddy water was studied in detail by Watanabe *et al*.(2006) who reported that an extension of water holding period to 10 days rather than 3 - 4 days after herbicide application for controlling herbicide runoff in paddy fields in Japan. In Kuttanadu, many new herbicides are introduced in recent years. It is essential to study the dissipation pattern of all the newly introduced herbicides and water holding period should be fixed and strictly advocated in order to avoid any possible contamination in the river systems.

The dissipation of chlorpyriphos in soil of Kuttanadu rice ecosystem is presented in Table 45. The data revealed that residues reached below the detectable level of 0.005 ppm on 15 DAS in the paddy field. The initial deposit of 0.561 ppm reached BDL after 15 DAS with a half life of 2.23 days. The residues of chlorpyriphos in the field outlet was lower than those existed in the field, the initial deposit being 0.165ppm which reached BDL in 4 DAS. In the drainage channel chlorpyriphos residues were observed at 48 HAS. Both in stream and river no residue was detected in all the occasions. Degradation of chlorpyriphos at faster rates in flooded condition than at field capacity moisture conditions in soils of Kerala was reported earlier by George *et al.* (2007).

The mean residues of chlorpyriphos in water in rice field were 0.32 ppm at 24 HAS and became BDL on 7 DAS. The chlorpyriphos residues were detected in field outlet when observed at 48 HAS and gradually declined to BDL on 5 DAS. Various factors that affect a pesticide's ability to be present in water include its water solubility, the distance from its application site to the body of water, weather and soil type, presence of growing crop and method of application of the chemical. All these factors might have influenced the presence of residues in water in the present study.

In a field study conducted in Malaysia, half life of chlorpyriphos was observed as 19.8 days in soil. In the present study the half life of chlorpyriphos was as low as 2.23 which may be due to the flooded conditions existing in the Kuttanadu rice eco-system. George *et al.* (2007) reported that degradation of chlorpyriphos is comparatively faster in flooded condition than at field capacity.

The dissipation of acephate in soil of Kuttanadu rice ecosystem is presented in Table 47. The data revealed that residues reached below the detectable level of 0.01 ppm on 72 HAS in the paddy field. The initial deposit of 0.0595 ppm reached BDL after 72HAS with a half life of 0.75 days. The residue of acephate was detected only in the paddy field. Residues in water samples were below the detectable level. The present study revealed that residues of acephate did not persist longer in wet land ecosystem compared to chlorpyriphos. Faster degradation of acephate with a half life of 0.4 to 2.6 days in soil was reported earlier. Lower persistence of polar pesticides like acephate in soil may be attributed to increased leaching loss and rapid degradation which was activated due to high moisture content of the soil.

The mean residues of hexaconazole in paddy soil were 0.359, 0.307, 0.162 and 0.106 ppm at 24, 48, 72 HAS and 4 DAS respectively. When analyzed at five and seven days after spraying the residues were found to be below detectable limit. The half life was worked out to be 1.36 days. Increased leaching loss and rapid degradation which was activated due to high moisture content of the soil might be responsible for the low half life observed in the present study. The mean residue in water at 48 and 72 HAS were 0.21 and 0.15 ppm respectively.
The dissipation of different pesticides are presented in Figure 12.

The dissipation of lambda cyhahothrin in rice plants in Kuttanadu rice ecosystem following application at 90 DAS is presented

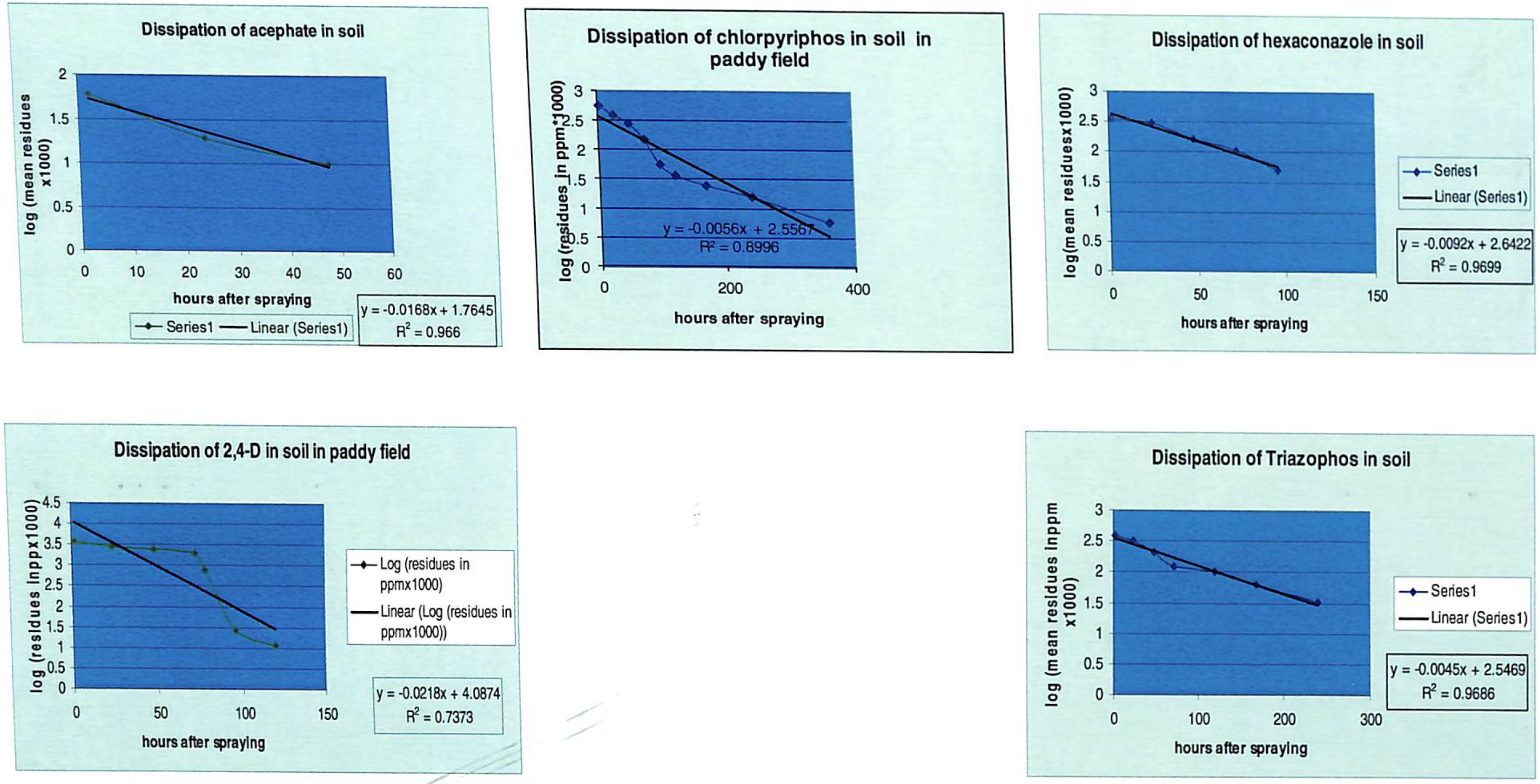
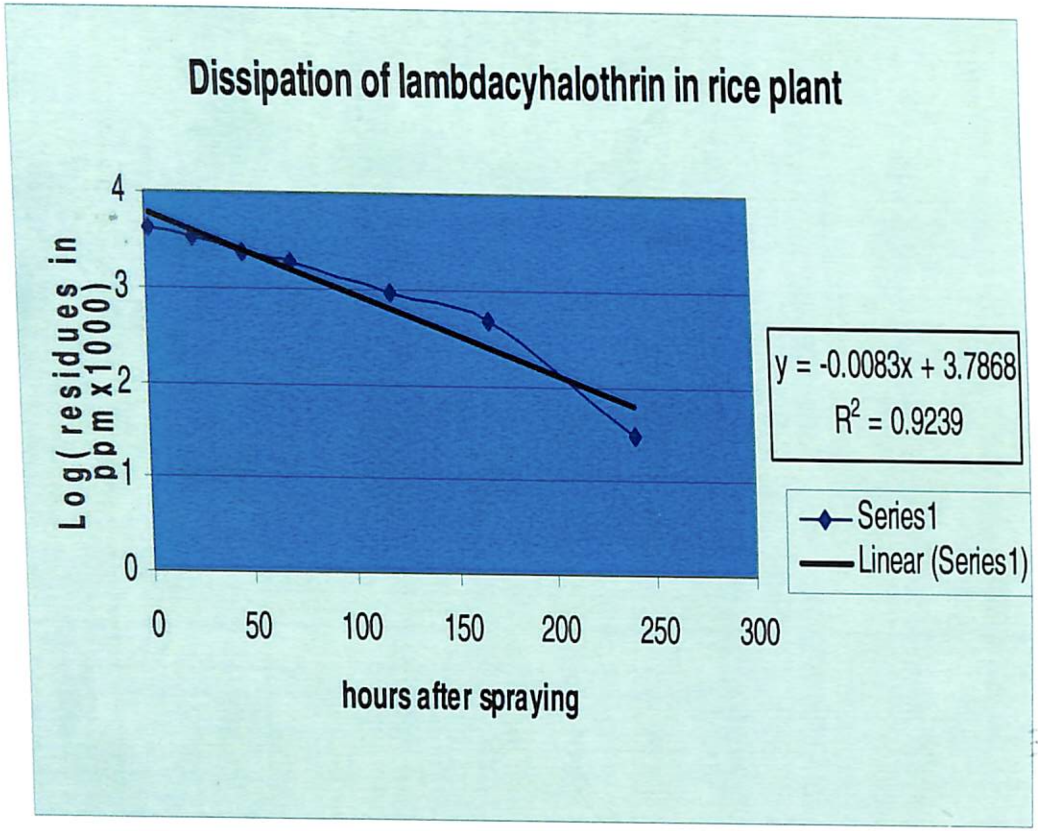


Figure 12. Dissipation of pesticides in soil in Kuttanad rice eco system

in fig.13. The data revealed that from an initial deposit of 4.10 ppm, the residues dissipated to 0.031 ppm by 10 DAS and reached BDL by 15 DAS with a half life of 1.51 days. Residues could not be seen in any of the soil or water samples. This may be due to the thick crop canopy towards the later stages of the crop which prevents the fall of the spray fluid in to the lower layers of soil and water. Further sunlight makes the break down of lambda-cyhalothrin faster in water and soil (Hornsby *et al*.,1995).

The dissipation of monocrotophos in soil of Kuttanadu rice ecosystem is presented in Table 53. The data revealed that residues reached below the detectable level of 0.01 ppm on 72 HAS in the paddy field. The initial deposit of 0.297 ppm reached BDL on 4DAS with a half life of 1.23 days. The residue of monocrotophos was detected in soils of in the paddy field only. The half life reported for monocrotophos in garden soil is higher than that observed in the present study which may be due to the rapid degradation under high moisture conditions. The mean level of residues in water sample in the rice field was 0.42 ppm which got reduced to 0.023 ppm at 72 HAS.

The mean residues of hexaconazole 0.359 ppm in soil at two HAS dissipated to 0.307 ppm at 24 HAS. The mean residues were 0.162 and 0.106 ppm respectively at 48 and 72 HAS. When analyzed five days and seven days after spraying the residues were found to be below detectable limit. The mean residues in water when observed at 48 HAS was 0.2091 ppm and when observed at 72 HAS was 0.1452 ppm. The mean residue of triazophos in paddy soil was 0.388 ppm at 24 HAS which dissipated to BDL by 15DAS, with a half life of 2.79



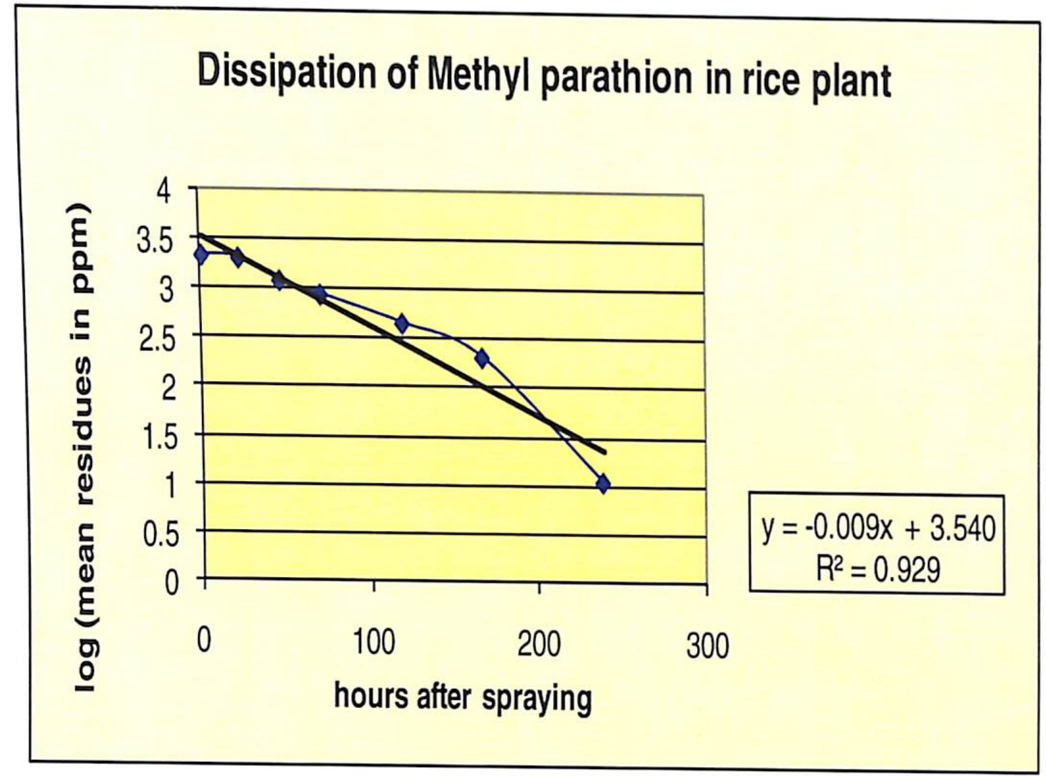


Figure 13. Dissipation of pesticides in rice plant in Kuttanad eco system

days. Though residues were detected in soil up to 15 DAS in water the residues reached BDL at 72 HAS.

The dissipation of methyl parathion in rice plants in Kuttanadu rice ecosystem following application at 90 DAS is presented in Table 54. The data revealed that from an initial deposit of 2.131 ppm, the residues dissipated to 0.011 ppm by 10 DAS and reached BDL by 15 DAS with a half life of 1.38 days. Similar half life values were reported by Seiber *et al* (1986) in flooded rice ecosystem. Residues could not be seen in any of the soil or water samples. This may be due to the thick crop canopy towards the later stages of the crop which restricts the fall of the spray fluid in to the lower layers of soil and water as in the case of lambda cyhalothrin applied in rice plants at 90 DAS.

5.6 Estimation of dermal exposure of spray men to pesticides during application of pesticides in Kuttanadu rice ecosystem

In order to evaluate the health hazard involved when working with toxic pesticides it is important to determine the amount of exposure that workers undergo while carrying out plant protection operations. There are three main routes of exposure, oral, inhalation and dermal. Most of the pesticides can be absorbed to some extent by all the three routes, but the formulation of a product has a large effect on potential absorption. As far as the spray men are concerned, the chances are more through dermal exposure. Drift during application also contribute

Table 60. Half life of pesticides in rice ecosystem

Pesticide	Matrix	Regression equation	R^2	Half –life (Days)
2,4-D	Soil exp I	Y= -0.0212X + 3.9314	0.9208	0.59
2,4-D	Soil exp II	Y= -0.0218X + 4.0874	0.7373	0.58
Chlorpyriphos	Soil	Y= -0.0056X + 2.5567	0.8996	2.24
Acephate	Soil	Y= -0.0168X + 1.7645	0.966	0.75
Hexaconazole	Soil	Y= -0.0092X + 2. 6422	0.969	1.36
Lamda cyhalothrin	Plant	Y= -0.0083X + 3.7868	0.923	1.51
Monocrotophos	Soil	Y= -0.0102X + 2. 5995	0.871	1.23
Triazophos	Soil	Y= -0.0045X + 2.5469	0.968	2.79
Methyl parathion	Plant	Y= -0.009X + 3.540	0.929	1.38

towards dermal exposure. In general, the hands and forearms receive the most exposure.

In the present study, the dermal exposure of hexaconazole to the uncovered portions of body of the spray men engaged on the operations at the tillering stage of paddy crop was 2.14 ± 0.109 $mg^{-1}m^{2}$ h^{-1}. The total dermal exposure of pesticide per man was calculated to be 3.76 ± 0.116 $mg^{-1}man^{-1}h^{-1}$. Similarly, the average dermal exposure per kg body weight per day was worked out to be 0.36 ± 0.011 mg. In the case of triazophos the corresponding values were 3.55 ± 0.131, 6.20 ± 0.152 and 0.59 ± 0.015 mg respectively. In a similar study in apple orchards, Durham and Wolfe (1962) reported a dermal exposure of 77.7 $mg^{-1}man^{-1}h^{-1}$ of methyl parathion to spray men. Comparatively low level of exposure recorded in the present study may be attributed towards the difference in crop canopy. The average dermal exposure per kg body weight per day of 0.36 mg and 0.59 mg for hexaconazole and triazophos were much lower than that reported by Fletcher *et al.* (1959) for dieldrin (1.8mg $kg^{-1}day^{-1}$). The use of personal protective devices (PPD) when spraying can reduce contact and inhalation of pesticides thereby potentially reducing the acute and chronic health hazards of pesticides to the spray men. However, more detailed studies have to be conducted to explore the health risk associated with the spray men during prolonged exposure to pesticides. The results of the present study as well as studies conducted on pesticide handling practices and pesticide exposure beliefs emphasize that there is a great need for pesticide safety education, which seems to be a universal problem in pesticide spraying.

The present study on “Impact of pesticides on biotic and abiotic components in Kuttanadu portray the present scenario of pesticide use, its implications on farmer health and the extent of contamination in food and environmental components in Kuttanadu. The results suggest the pesticides use in rice fields of Kuttanadu do not represent a significant environmental threat by way of contamination of non target sites of application such as drainage channels, streams or river in the region. The present conclusion drawn based on the studies covering one or two crop seasons need not be taken as thumb rule. However, extensive studies for prolonged periods have to be taken up to get conclusive scenario of the region. Since the wet lands of Kuttanadu are “washed off” every year by sea water inundation, the chance of cumulative effect of pesticides on environment is meager. The present study emphasis the importance of water holding period after pesticide application in order to minimize the possible contamination to adjacent water bodies. The water holding period should be prescribed for every pesticide especially for herbicides and should be strictly followed. The study highlight the adverse effects of unscientific pesticide handling and disposal practices followed by the farmers in Kuttanadu eco-system depicts total ignorance of the farmers towards Good Agricultural

Practices in pesticide use and warrants the effective implementation of proper training. Further, the situation demands immediate policy decisions and strict interventions for legal enforcement of FAO guide lines on safe pesticide use in the region. It can be concluded that it is the need of the hour that we should concentrate more on pesticide related externalities which have negative effect on farming community resulting from the impaired farmer health due to direct and indirect exposure to pesticides.

SUMMARY

6. SUMMARY

Rice, the major crop raised in Kuttanadu, is prone to infestation by several insect pests, diseases and weeds which necessitate the use of pesticides for their timely control. Rice being grown under submerged soil condition, the pesticides applied directly can reach the field water which will be pumped out to canals, streams, rivers, lakes etc. Pesticides applied to the rice crop may persist in the environment and contaminate the produce with their toxic residues. There is an urgent need to bring out the current status of pesticide contamination of abiotic and biotic components of rice ecosystem of Kuttanadu and help in developing measures to minimize the residue hazards. Hence an extensive study was carried out in the rice eco system of Kuttanadu during 2007-2010 and the salient findings are summarized below.

- Survey conducted on the pesticide use and consumption pattern in the three river catchments in Kuttanadu *viz.,* Pampa, Manimala and Meenachil revealed that the herbicide use in Kuttanadu has increased during the past five years while a decreasing trend was noticed the case of insecticides.
- The average consumption of pesticides in Kuttanadu ranged from 0.56 to 3.38 kg ai ha^{-1} with 0.3 to 1.63, 0.04 to 0.097 and

0.09 to 0.947 kg ai $^{-1}$ for herbicides, fungicides and insecticides respectively.

- Out of the 23 popular pesticides in Kuttanadu, herbicides and fungicides constitute 18.18 per cent each and insecticides constitute 65.22 per cent. The most popular herbicide, fungicide and insecticide were 2, 4-D, hexaconazole and acephate respectively.
- Pesticide dealers or representatives acted as the main source of information to the farmers regarding pesticide use. Categorization of the different pesticides used in the region in terms of toxicity revealed that 8.70 per cent belonged to the group 'less toxic' with green colour code, 39.13 per cent each belonged to the group 'moderately toxic' with blue colour code, and to the group 'highly toxic' with yellow colour code and 17.39 per cent to 'extremely toxic' with red colour code.
- The survey revealed that 75 per cent of the farmers are aware of the concept of IPM though the extent of adoption is only 39.16%. However, the awareness of Good Agricultural Practices among the farmers is only 4 per cent.
- The survey further revealed that during pesticides application, the spray men were not using any protective gadgets as per FAO

guidelines while 18 per cent of the spray men were using partial protective measures.

- The label information on the pesticide as well as the safety guidelines for disposal of used pesticide bottles and wash water of sprayer were not followed by the majority of farmers.
- Health problems like head ache, dizziness, skin irritation etc were reported by the spray men.
- For monitoring of pesticide residues in various components in the rice ecosystem, multi residue methods in water, soil, paddy grains, paddy straw, duck meat and fish were developed and validated Five important validation parameters *viz.,* recovery percentage, repeatability, reproducibility, linearity, limit of detection and limit of quantification were established for eleven candidate pesticides *viz.,* phorate, dimethoate, lindane, methyl parathion, malathion, chlorpyriphos, alpha endosulfan, hexaconazole, ediphenphos, lambda cyhalothrin and deltamethrin. These methods were found to be suitable for estimating multiple pesticide residues in different components of Kuttanadu rice ecosystem. The estimation of residues from different components were performed using gas chromatograph (GC) and high performance liquid chromatograph(HPLC).

- Monitoring of pesticide residues in various components were conducted in three different river catchments in Kuttanadu. Samples of soil and water were collected from rice field, field outlet, drainage channel, stream and river at different growth stages of the crop viz., seedling stage, tillering stage, booting stage, milky stage and after the harvest. Samples of rice grains, straw, fish, mollusk, duck meat, eggs and animal meat (beef) were collected from the rice ecosystem. Studies revealed the presence of pesticides like 2,4-D, chlorpyriphos, dimethoate, phorate, phorate sulphone, carbaryl, triazophos, quinalphos, monocrotophos and malathion.
- The residues of 2,4-D in water samples ranged from 0.01 to 0.056 ppm during the seedling stage. The pesticides detected during the tillering stage were monocrotophos (0.01 to 0.03 ppm), chlorpyriphos (0.005 to 0.02 ppm), triazophos (0.01 to 0.03 ppm) and phorate (0.038 to 0.062). Residue of malathion (0.01 to 0.05 ppm) was detected in water samples during the booting stage.
- The residues of 2,4-D (0.018 to 0.2 ppm), dimethoate (0.01 to 0.3) were detected in soil samples during seedling stage. During the tillering stage residues of chlorpyriphos (0.1 to 0.32 ppm), triazophos (0.0.2 to 0.32 ppm), quinalphos (0.05 ppm), carbaryl (0.32 ppm) and phorate sulphone (0.502ppm) were detected.

The pesticide residues were below detectable limit in samples of rice grains, straw, fish, duck meat, eggs and beef collected from the rice ecosystem.

- Studies conducted on the impact of IPM and conventional practices on major pests, natural enemies and neutrals revealed that during the initial stages (25DAS), the pest : defender ratio was 1.031 and 1.141 in the IPM and conventional plots, while at 45 DAS the pest: defender (P:D) ratio was 1.836 and 2.118 respectively. At 65 DAS the P: D ratio was 1.763 and 3.037 respectively in IPM and conventional plots. The mean yield of paddy grains and straw were higher in IPM plots.
- Studies conducted on the field experiment on the dissipation of important pesticides being used in Kuttanadu revealed that the dissipation in field conditions was mainly influenced by the water management practices prevailing in the region. The half life of 2,4-D, chlorpyriphos, acephate, hexaconazole, moncrotophos and triazophos in soil were found to be 0.59, 2.24, 0.75, 1.36, 1.23, 2.79 days respectively.
- The half life of lambda cyhalothrin and methyl parathion in rice plants were 1.51 and 1.38 days respectively.
- The present studies revealed that appreciable quantity of pesticide residues were present in the field and field outlet

while the quantity of pesticide residues present in the drainage channel was low.

- The pesticide residues in river and stream were below detectable limit.
- Estimation of dermal exposure of spray men to pesticides during application of pesticides in Kuttanadu rice ecosystem revealed that the dermal exposure of hexaconazole and triazophos per man were calculated to be 3.76 $\pm$0.116 mg $man^{-1}h^{-1}$,6.20 $\pm$0.152 mg $man^{-1}h^{-1}$respectively.

On basis of the present investigation it can be concluded that the apprehension on the contamination of water bodies in Kuttanadu rice ecosystem is not that serious as conceived by the public. However, for a meaningful interpretation of the present findings, greater research support have to be explored and an intensive study covering the entire Kuttanadu rice ecosystem have to be taken up to arrive at definite conclusion.

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IMPACT OF PESTICIDES ON ABIOTIC AND BIOTIC COMPONENTS IN RICE ECOSYSTEM OF KUTTANADU

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Abstract of the thesis submitted in partial fulfillment of the requirement for the degree of

DOCTOR OF PHILOSOPHY IN AGRICULTURE

Faculty of Agriculture

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ABSTRACT

Investigations were carried out in the main crop seasons of Kuttanadu during the years 2007-08 and 2008-09 to study the pesticide use pattern and extent of contamination in rice ecosystem of Kuttanadu, to assess the relative impact of conventional and IPM practices and to suggest measures for minimizing the residue hazards.

The survey conducted on the pesticide use and consumption pattern in the three river catchments in Kuttanadu *viz.,* Pampa, Manimala and Meenachil revealed that the herbicide use in Kuttanadu has increased during the past five years while a decreasing trend was noticed the case of insecticides. The consumption of pesticides in Kuttanadu ranged from 0.56 to 3.38 kg ai ha^{-1} with 0.3 to 1.63, 0.04 to 0.0973 and 0.09 to 0.947 kg ai $^{-1}$ for herbicides, fungicides and insecticides respectively. The most popular herbicide, fungicide and insecticide were 2,4-D, hexaconazole and acephate respectively. The survey further revealed that during pesticides application, the spray men were not using any protective gadgets as per FAO guidelines while 18 per cent of the spray men were using partial protective measures. The label information on the pesticides as well as the safety guidelines for disposal of used pesticide bottles and wash water of sprayer were not followed by the majority of farmers. Health problems like head ache and dizziness, skin irritation etc were reported by the spray men.

Multi Residue Methods in water, soil, paddy grains, paddy straw, duck meat and fish were developed and validated by conducting recovery studies. Five important validation parameters *viz.,* recovery per cent age, repeatability, reproducibility, linearity, limit of detection and limit of quantification were established for eleven candidate pesticides *viz.,* phorate, dimethoate, lindane, methyl parathion, malathion, chlorpyriphos, alpha endosulfan, hexaconazole, ediphenphos, lambda cyhalothrin and deltamethrin. These methods were found to be suitable for analyzing multiple pesticide residues in different components of Kuttanadu rice ecosystem. The estimation of residues from different components were performed using gas chromatograph (GC) and high performance liquid chromatograph (HPLC).

Monitoring studies were conducted in three different river catchments in Kuttanadu. Samples of soil and water were collected from rice field, field outlet, drainage channel, stream and river at different growth stages of the crop viz., seedling stage, tillering stage, booting stage, milky stage and after the harvest. Samples of rice grains, straw, fish, mollusk, duck meat, eggs and animal meat (beef) were collected from the rice ecosystem. Data revealed that the pesticides detected included 2,4-D, chlorpyriphos, dimethoate, phorate, phorate sulphone, carbaryl, triazophos,monocrotophos, quinalphos and malathion. The pesticide residues were below detectable limit in samples of rice grains, straw, fish,mollusk, duck meat, eggs and beef collected from the rice ecosystem.

Studies conducted on the impact of IPM and conventional practices on major pests, natural enemies and neutrals revealed that during the initial stages (25DAS), the pest : defender ratio was 1.031 and 1.141 in the IPM and conventional plots, while at 45 DAS the pest: defender (P:D) ratio was 1.836 and 2.118 respectively. At 65 DAS the P:D ratio was 1.763 and 3.037 respectively in IPM and conventional plots. The mean yield of paddy grains and straw were higher in IPM plots.

Studies on the dissipation of pesticides being used in Kuttanadu revealed that the dissipation in field conditions was mainly influenced by the water management practices prevailing in the region. The half life of 2,4-D, chlorpyriphos, acephate, hexaconazole, moncrotophos and triazophos in soil were found to be 0.59, 2.24, 0.75, 1.36,1.23 and 2.79 days respectively. The half life of lambda cyhalothrin and methyl parathion in rice plants were 1.51 and 1.38 days respectively. The present studies revealed that appreciable quantity of pesticide residues were present in the field and field outlet while the quantity of pesticide residues present in the drainage channel was low. The pesticide residues in river and stream were below detectable limit.

Estimation of dermal exposure of spray men to pesticides during application of pesticides in Kuttanadu rice ecosystem revealed that the dermal exposure of hexaconazole and triazophos per man were calculated to be 3.76 $\pm$0.116 mg $man^{-1}h^{-1}$, 6.20 $\pm$0.152 mg $man^{-1}h^{-1}$respectively.

The study emphasizes the need for extensive study to be conducted at compartmental level to assess the exact route of dissipation of the pesticides applied and to arrive at the extent of environmental impact due to their application. A safe pest control strategy envisaging the use of pesticides need to be developed and recommended based on the detailed study in the ecosystem. The farmers and the workers are to be trained properly so as to follow judicious plant protection procedures to ensure Good Agricultural Practices in rice cultivation in the region without disturbing the natural eco system for sustainable development.

APPENDICES

CHROMATOGRAMS OF MIXTURE OF PESTCIDES

AINP ON PESTICIDE RESIDUES

CSS on MPR at National Level, College of Agriculture

Vellayani, Thiruvanathapuram

Sample Information

User Name : Admin
Sample Name : FAO mix ABCD 0.1ppm
Sample ID : FAO mix ABCD 0.1ppm
Sample Type : Standard
Injection Volume : 1.00
Sample Amount : 1
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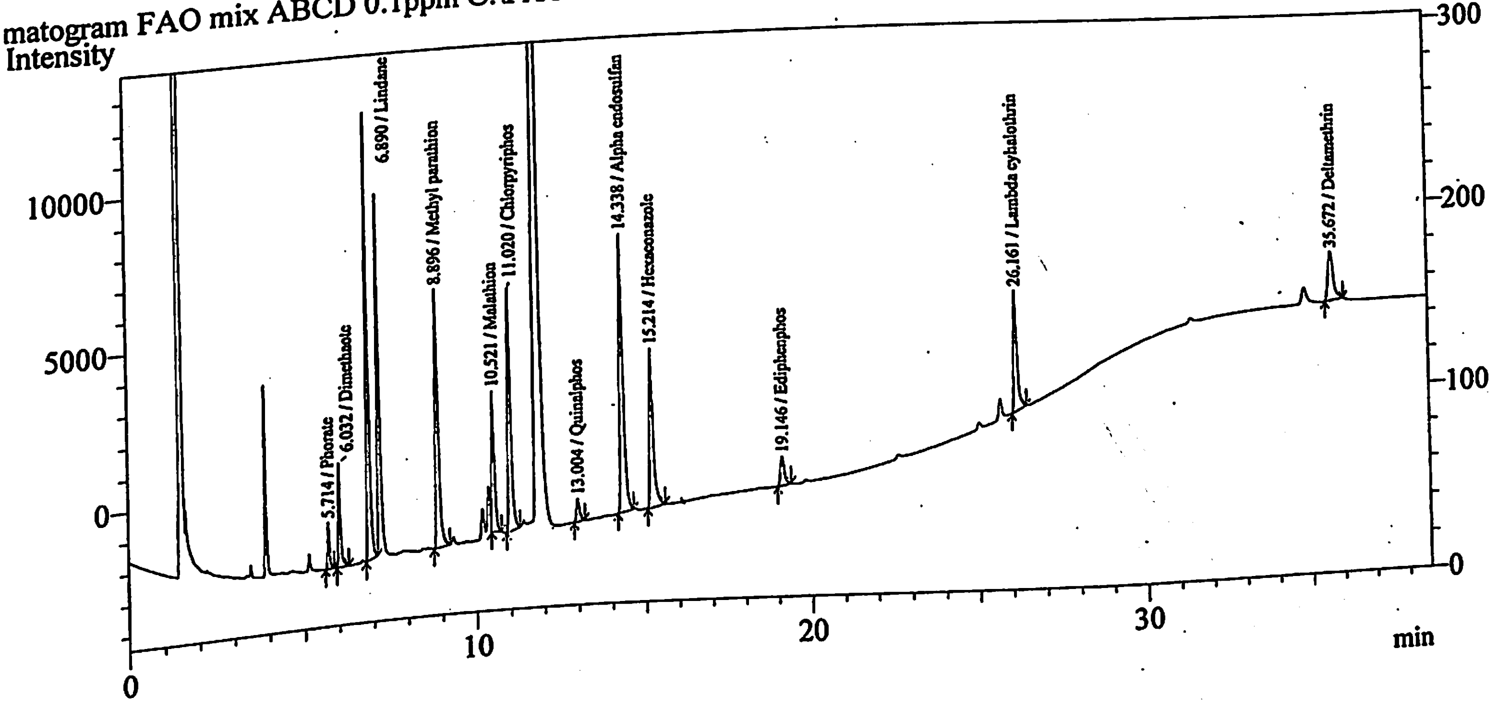
Analysis Date & Time : 5/23/2009 2:15:14 PM



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Units	Name
1	5.714	5809	1505	ppm	Phorate
2	6.032	14511	3341	ppm	Dimethaote
3	6.890	62627	14451	ppm	Lindane
4	8.896	45997	8395	ppm	Methyl parathion
5	10.521	25063	4543	ppm	Malathion
6	11.020	46323	7873	ppm	Chlorpyriphos
7	13.004	4556	739	ppm	Quinalphos
8	14.338	60948	9015	ppm	Alpha endosulfan
9	15.214	36846	5120	ppm	Hexaconazole
10	19.146	7211	917	ppm	Ediphenphos
11	26.161	26167	3944	ppm	Lambda cyhalothrin
12	35.672	18029	1604	ppm	Deltamethrin

AINP ON PESTICIDE RESIDUES

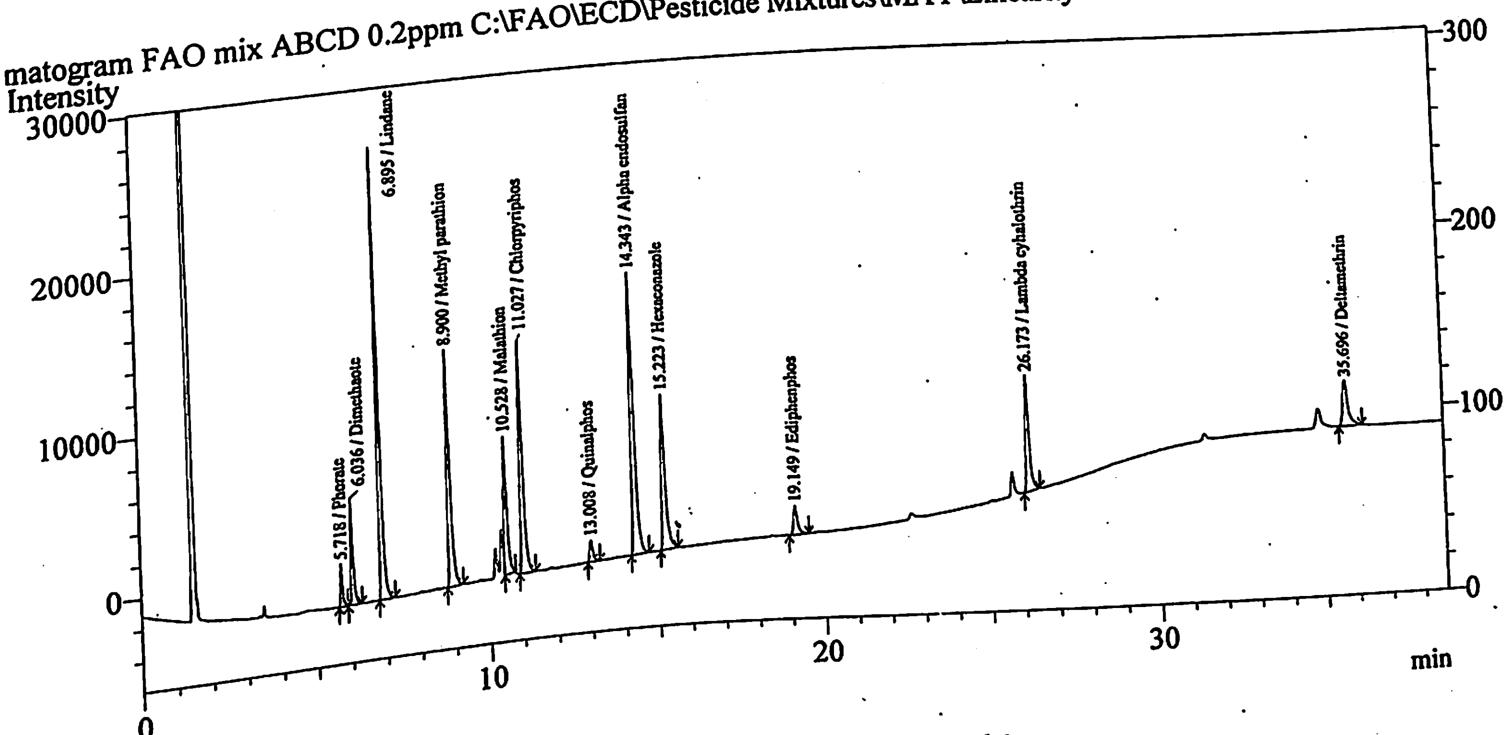
CSS on MPR at National Level, College of Agriculture

Vellayani, Thiruvanathapuram

Sample Information

User Name : Admin
Sample Name : FAO mix ABCD 0.2ppm
Sample ID : FAO mix ABCD 0.2ppm
Sample Type : Standard
Injection Volume : 1.00
Sample Amount : 1
Method Name : C:\FAO\ECD\Pesticide Mixtures\MAY\Linearity\FAO Mix ABCD linearity.gcm
Analysis Date & Time : 5/23/2009 1:32:03 PM

C:\FAO\ECD\Pesticide Mixtures\MAY\Linearity\FAO mix ABCD 0.2ppm.gcd - Chan
matogram FAO mix ABCD 0.2ppm C:\FAO\ECD\Pesticide Mixtures\MAY\Linearity\FAO mix ABCD 0.2ppm.gcd - Chan



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Units	Name
1	5.718	10858	2714	ppm	Phorate
2	6.036	29467	6703	ppm	Dimethaote
3	6.895	128634	28590	ppm	Lindane
4	8.900	83691	14944	ppm	Methyl parathion
5	10.528	49609	8691	ppm	Malathion
6	11.027	87517	14681	ppm	Chlorpyriphos
7	13.008	8825	1365	ppm	Quinalphos
8	14.343	119630	17997	ppm	Alpha endosulfan
9	15.223	73545	9820	ppm	Hexaconazole
10	19.149	15156	1795	ppm	Ediphenphos
11	26.173	48150	7327	ppm	Lambda cyhalothrin
12	35.696	33932	2910	ppm	Deltamethrin

AINP ON PESTICIDE RESIDUES

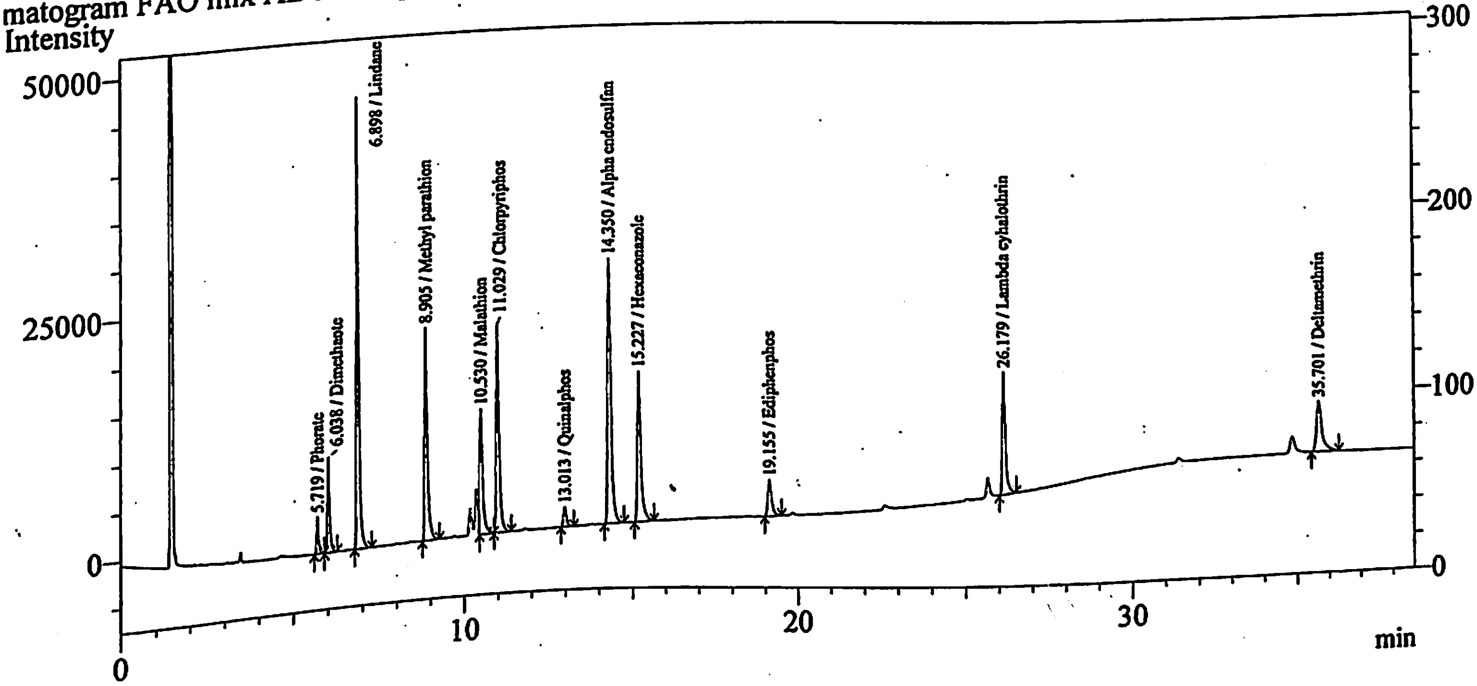
CSS on MPR at National Level, College of Agriculture

Vellayani, Thiruvanathapuram

Sample Information

User Name	: Admin
Sample Name	: FAO mix ABCD 0.3ppm
Sample ID	: FAO mix ABCD 0.3ppm
Sample Type	: Standard
Injection Volume	: 1.00
Sample Amount	: 1
Method Name	: C:\FAO\ECD\Pesticide Mixtures\MAY\Linearity\FAO Mix ABCD linearity.gcm
Analysis Date & Time	: 5/23/2009 12:48:56 PM

matogram FAO mix ABCD 0.3ppm C:\FAO\ECD\Pesticide Mixtures\MAY\Linearity\FAO mix ABCD 0.3ppm.gcd - Chan



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Units	Name
1	5.719	16181	4010	ppm	Phorate
2	6.038	44509	10094	ppm	Dimethaote
3	6.898	210123	47511	ppm	Lindane
4	8.905	125411	22521	ppm	Methyl parathion
5	10.530	82705	13409	ppm	Malathion
6	11.029	133518	22064	ppm	Chlorpyriphos
7	13.013	13477	2115	ppm	Quinalphos
8	14.350	188588	27961	ppm	Alpha endosulfan
9	15.227	115248	15979	ppm	Hexaconazole
10	19.155	30501	3878	ppm	Ediphenphos
11	26.179	83891	13074	ppm	Lambda cyhalothrin
12	35.701	63324	5464	ppm	Deltamethrin

AINP ON PESTICIDE RESIDUES

CSS on MPR at National Level, College of Agriculture

Vellayani, Thiruvanathapuram

Sample Information

User Name	: Admin
Sample Name	: MIX ABCD 0.5ppm 230509
Sample ID	: MIX ABCD 0.5ppm 230509
Sample Type	: Standard
Injection Volume	: 1.00
Sample Amount	: 1
Method Name	: C:\FAO\ECD\Pesticide Mixtures\MAY\Linearity\FAO Mix ABCD linearity.gcm
Analysis Date & Time	: 5/23/2009 11:06:18 AM

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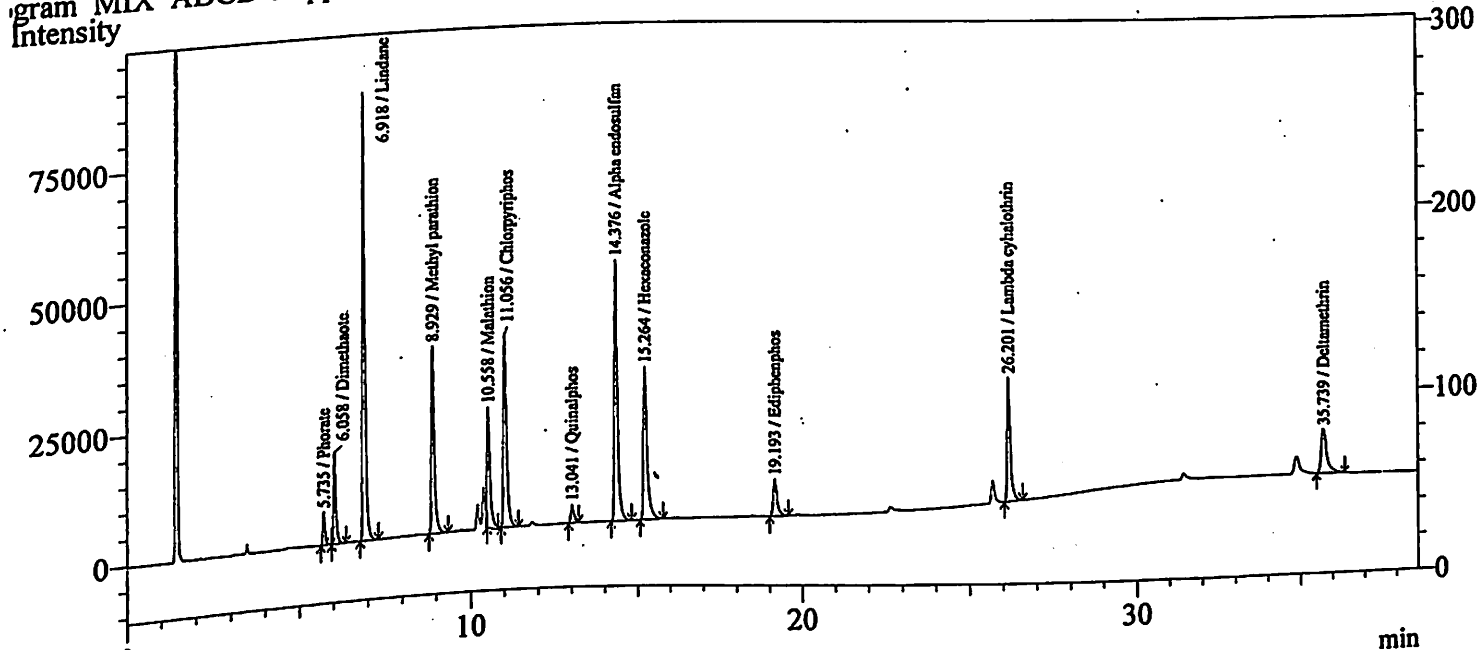
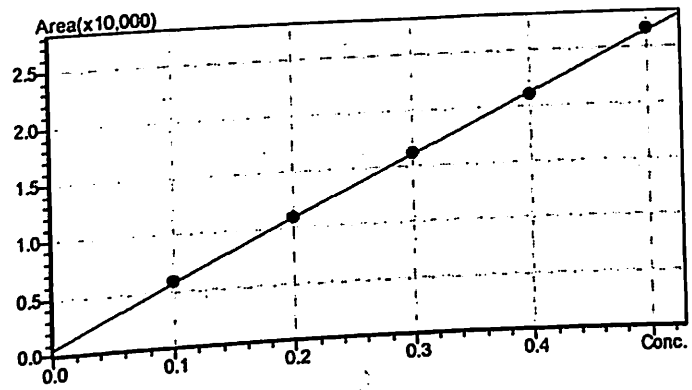


Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Units	Name
1	5.735	26604	6488	ppm	Phorate
2	6.058	78955	17751	ppm	Dimethaote
3	6.918	390465	86272	ppm	Lindane
4	8.929	207909	36233	ppm	Methyl parathion
5	10.558	140656	23317	ppm	Malathion
6	11.056	226405	37275	ppm	Chlorpyriphos
7	13.041	21359	3483	ppm	Quinalphos
8	14.376	342162	50078	ppm	Alpha endosulfan
9	15.264	224804	29487	ppm	Hexaconazole
10	19.193	59199	7183	ppm	Ediphenphos
11	26.201	153515	24089	ppm	Lambda cyhalothrin
12	35.739	104562	8720	ppm	Deltamethrin

LINEARITY CURVE OF PESTICIDES

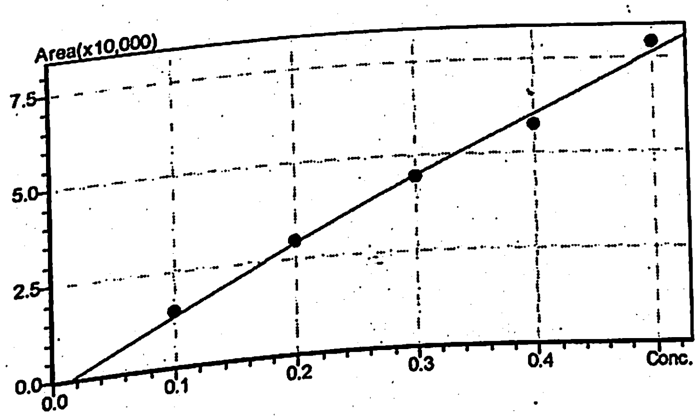
PHORATE



Level (ppm)	Area
0.1	5809
0.2	10858
0.3	16181
0.4	21025
0.5	26604

Y = aX + b a = 51807.46 b = 563.0371 R^2 = 0.9997090 R = 0.9998545
Mean RF : 54442.15 RF SD : 2133.688 RF %RSD : 3.919183

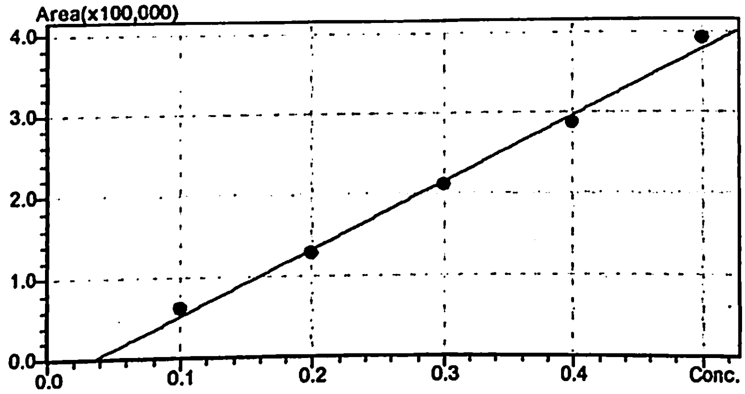
DIMETHAOTE



Level (ppm)	Area
0.1	14511
0.2	29467
0.3	44509
0.4	57336
0.5	78985

Y = aX + b a = 156955.0 b = -2090.849 R^2 = 0.9928916 R = 0.9964394
Mean RF : 148512.5 RF SD : 5547.405 RF %RSD : 3.735311

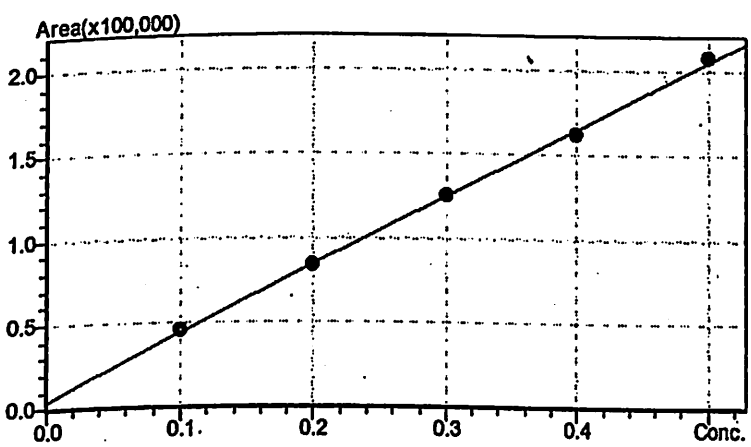
LINDANE



Level (ppm)	Area
0.1	62627
0.2	128634
0.3	210123
0.4	285915
0.5	390465

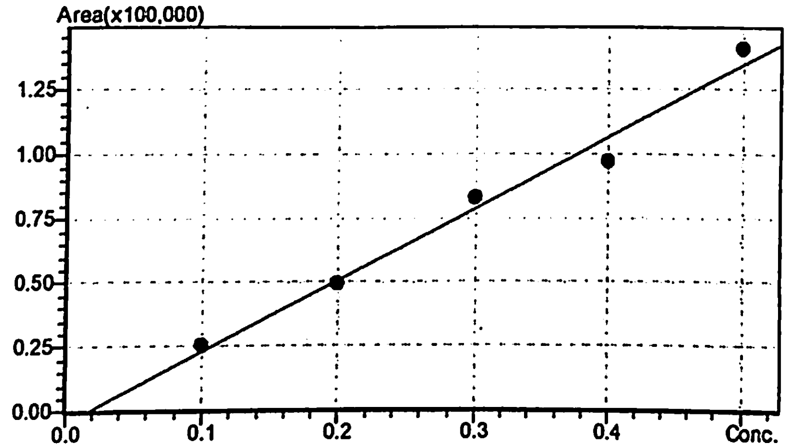
Y = aX + b a = 812956.6 b = -28334.33 R^2 = 0.9935972 R = 0.9967934
Mean RF : 693113.2 RF SD : 61638.03 RF %RSD : 8.892924

METHYL PARATHION



Level (ppm)	Area
0.1	45997
0.2	83601
0.3	125411
0.4	161655
0.5	207909

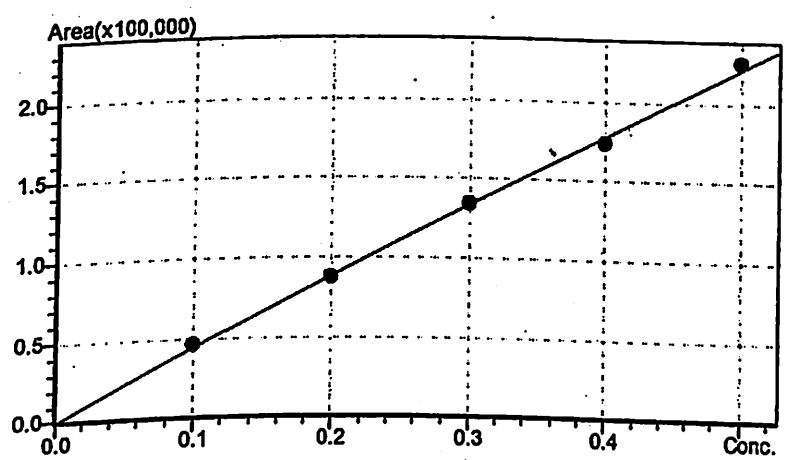
Y = aX + b a = 401783.5 b = 4397.112 R^2 = 0.9986263 R = 0.9993129
Mean RF : 423283.0 RF SD : 21327.86 RF %RSD : 5.038676



Level (ppm)	Area
0.1	25063
0.2	49609
0.3	82709
0.4	97361
0.5	140656

Y = aX + b a = 278937.5 b = -4602.41 R^2 = 0.9817441 R = 0.9908300
Mean RF : 259814.8 RF SD : 17365.41 RF %RSD : 6.683764

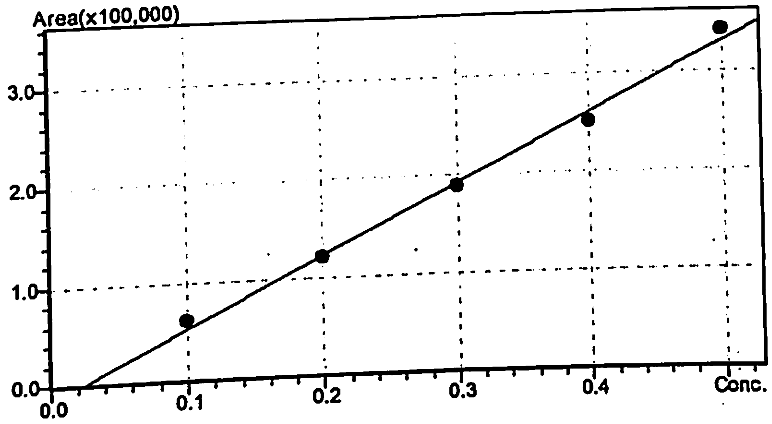
CHLORPYRIPHOS



Level (ppm)	Area
0.1	46323
0.2	87517
0.3	133518
0.4	123466
0.5	226405

Y = aX + ba = 446114.3 b = -388.4459 R^2 = 0.9979349 R = 0.9989669
Mean RF : 446469.5 RF SD : 11893.31 RF %RSD : 2.663857

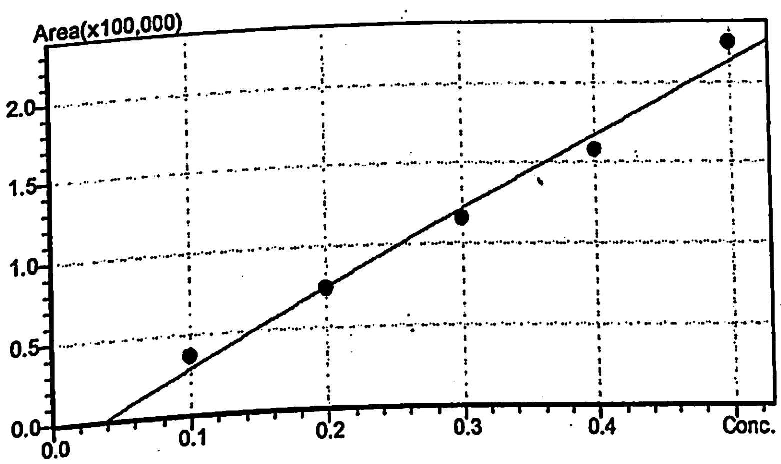
ALPHA ENDOSULFAN



Level (ppm)	Area
0.1	60948
0.2	119630
0.3	188588
0.4	251952
0.5	342162

Y = aX + b a = 694749.6 b = -15768.85 R^2 = 0.9938914 R = 0.9969410
Mean RF : 630092.7 RF SD : 33122.29 RF %RSD : 5.256734

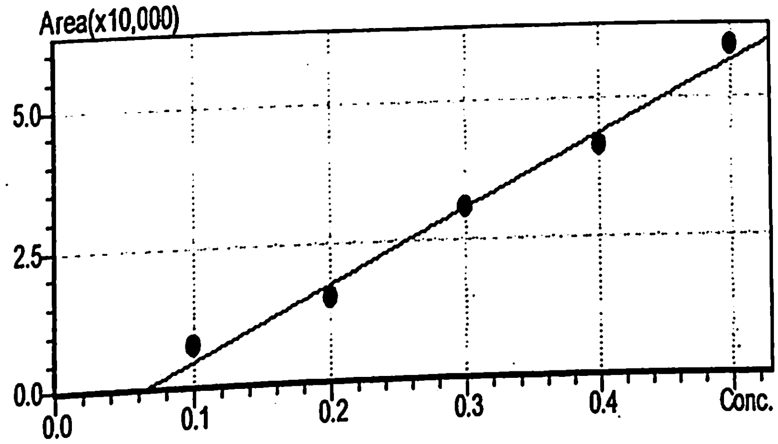
HEXACONAZOLE



Level (ppm)	Area
0.1	36846
0.2	73545
0.3	115248
0.4	157810
0.5	224804

Y = aX + b a = 460180.7 b = -16403.59 R^2 = 0.9851862 R = 0.9925655
Mean RF : 392895.6 RF SD : 33637.36 RF %RSD : 8.561398

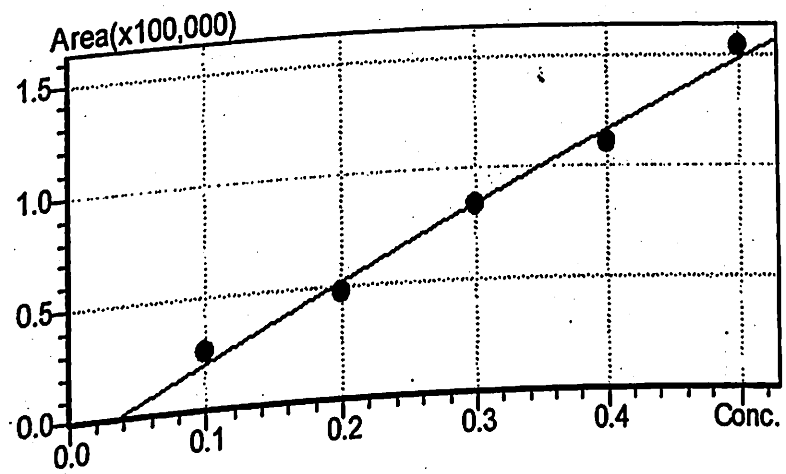
EDIPHENPHOS



Level (ppm)	Area
0.1	7211
0.2	15156
0.3	30501
0.4	40902
0.5	49199

Y = aX + b a = 129723.4 b = -8323.267 R^2 = 0.9843076 R = 0.9921228
Mean RF : 94041.88 RF SD : 19580.43 RF %RSD : 20.82096

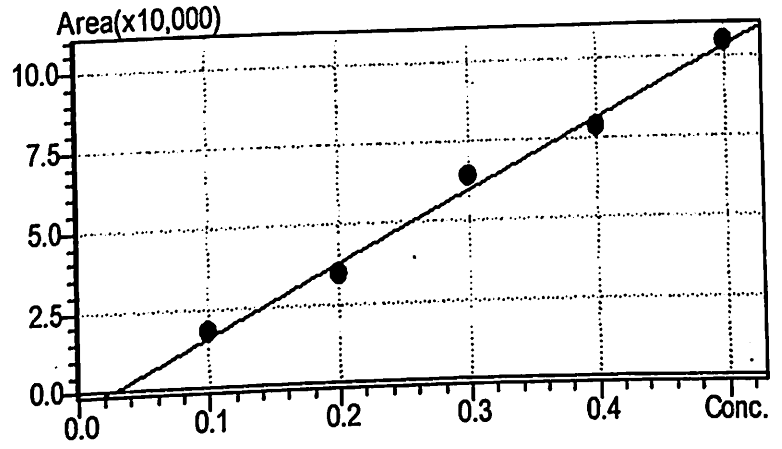
LAMBDA CYHALOTHRIN



Level (ppm)	Area
0.1	26167
0.2	48150
0.3	83899
0.4	110470
0.5	153515

Y = aX + b a = 317015.4 b = -10665.95 R^2 = 0.9889780 R = 0.9944737
Mean RF : 273052.8 RF SD : 24394.79 RF %RSD : 8.934092

DELTAMETHRIN



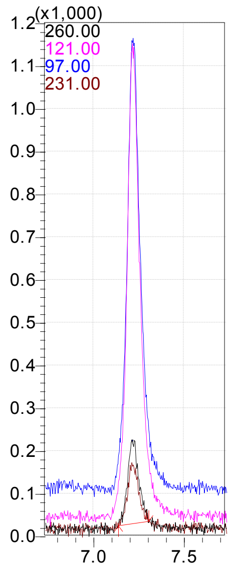
Level (ppm)	Area
0.1	18029
0.2	33932
0.3	63324
0.4	78586
0.5	104562

Y = aX + b a = 217719.0 b = -5629.068 R^2 = 0.9910781 R = 0.9955291
Mean RF : 193324.5 RF SD : 18058.33 RF %RSD : 9.340942

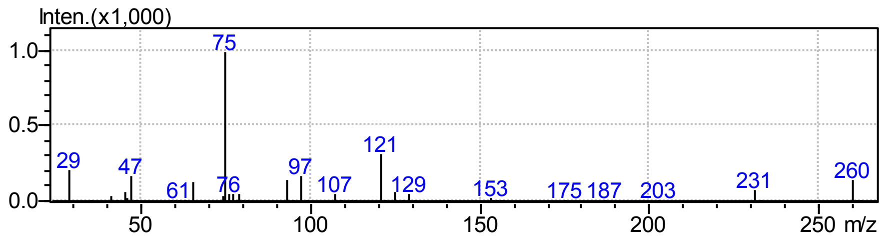
GC-MS CHROMATOGRAMS

APPENDICES

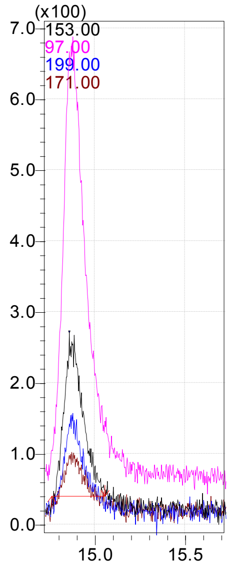
MS Chromatogram (Selective Ion Monitoring Mode)
Phorate 1 ppm RT 7.220



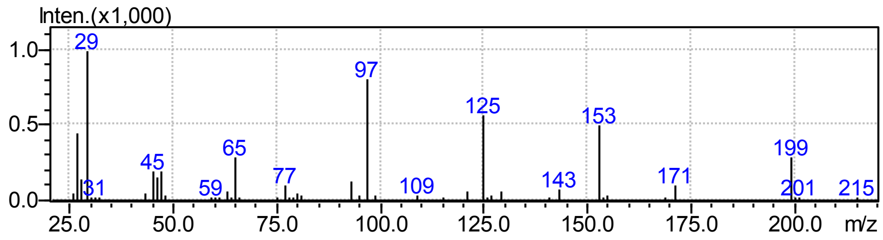
Mass spectrum of Phorate



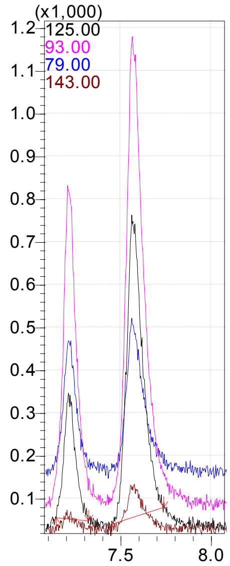
MS Chromatogram (Selective Ion Monitoring Mode)
Phorate sulfone 1 ppm RT 15.225



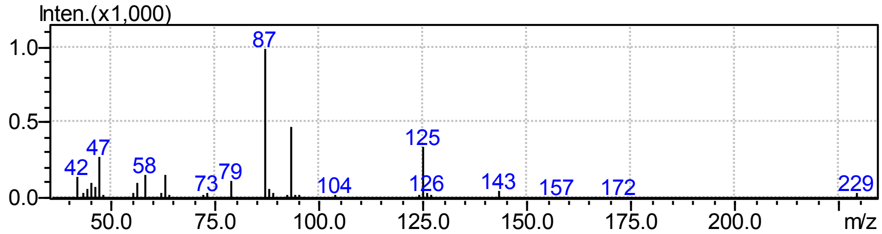
Mass spectrum of Phorate Sulphone



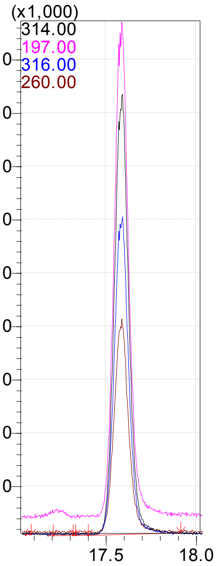
MS Chromatogram (Selective Ion Monitoring Mode)
Dimethoate 1 ppm RT 7.580



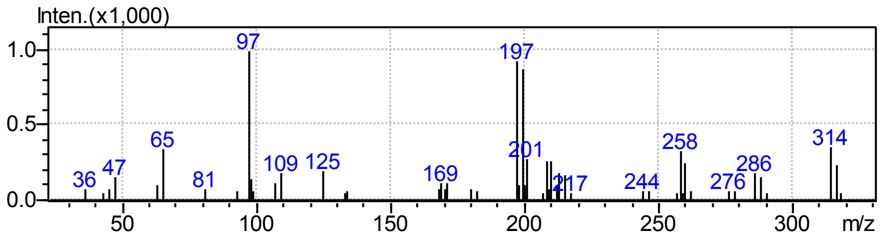
Mass spectrum of Dimethoate



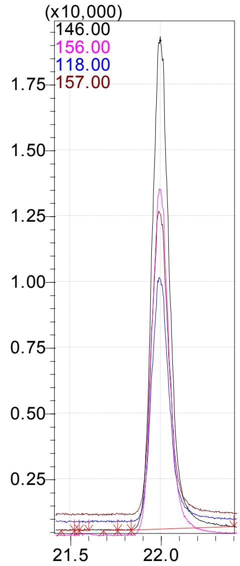
MS Chromatogram (Selective Ion Monitoring Mode)
Chlorpyrifos 0.5 ppm RT 17.533 minutes



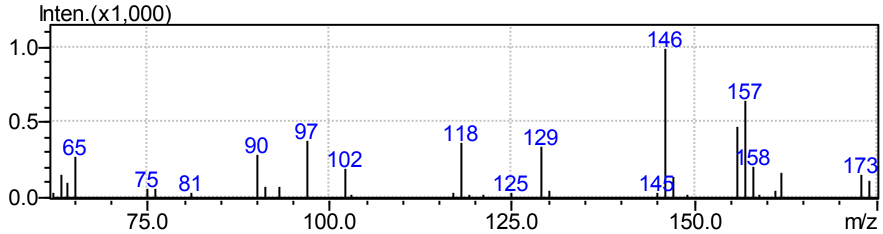
Mass spectrum of Chlorpyrifos



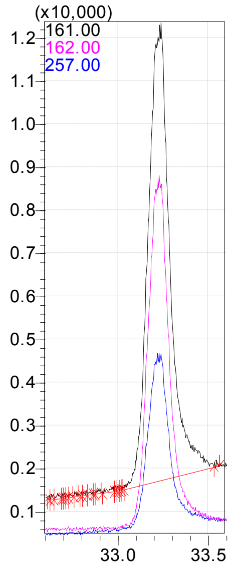
MS Chromatogram (Selective Ion Monitoring Mode)
Quinalphos 0.5ppm RT 21.917 minutes



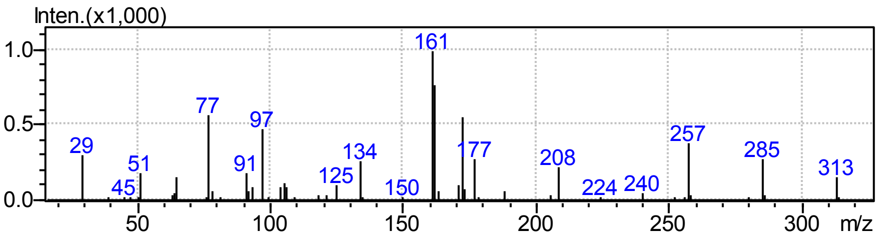
Mass spectrum of Quinalphos



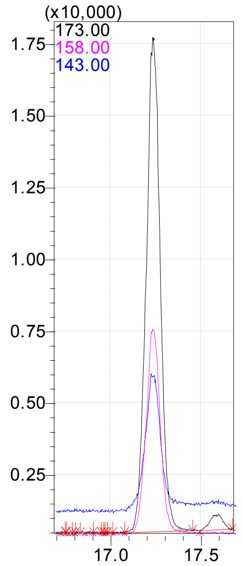
MS Chromatogram (Selective Ion Monitoring Mode)
Triazophos 0.5ppm RT.33.100 minutes



Mass spectrum of Triazophos



MS Chromatogram (Selective Ion Monitoring Mode)
Malathion 0.5 ppm RT 17.192 minutes



Mass spectrum of Malathion