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RESIDUES OF CHLORPYRIPHOS AND MONOCROTOPHOS IN SOIL AND WATER OF OTTANCHATHARAM BLOCK, INDIA

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ABSTRACT

The indiscriminate use of pesticides to meet the demand of growing population led to environmental contamination and consequently health risks. The study was undertaken to assess residue levels in vegetable growing area of Ottanchathiram block of Dindigul District. The study revealed that about 55 per cent the farmers applied 4 kg or less of technical grade pesticides per ha and the remaining 45 per cent used more than 4 kg of active ingredient (a.i)/ ha in chillies and About 87 per cent of cauliflower growers applied pesticides amounting to 4 kg or less of a.i/ha and the remaining applied more than this quantity. The villages, Thasaraipatti, Veeralapatti,Ambilikai, Arasappapillaipatti, Vadakadu, Kosavapatti, Krishnakoundanputhur and Virupachi are intensive vegetable growing areas selected for the pesticide residue analysis. The village Thasaraipatti soil sample recorded maximum amount of Chlorpyriphos residues (5.6 µgkg^{-1}) and Monocrotophos residues (4.5µgkg^{-1}) Residues in water samples ranged from not detectable to 0.5 µgl^{-1} of Chlorpyriphos and Monocrophos

KEY WORDS : Survey-pesticide use, Chlorpyriphos, Monocrotophos, Residues-soil water

INTRODUCTION

The term pesticide covers a wide range of compounds including insecticides, fungicides, herbicides, rodenticides, molluscicides, nematicides, plant growth regulators and others. These pesticides can be generally classified into four main chemical groups: organophosphate (OP), organochlorines (OC), carbamates/ dithiocarmates (EBDC), and synthetic pyrethroids. About 5.2 billion Pounds of pesticides used worldwide (EPA 2009). In India, the production of pesticides started in 1952 with the establishment of a plant for the production of BHC near Calcutta, and India is now the second largest manufacturer of pesticides in Asia after China and ranks twelfth globally. In India the Registration Committee (RC) has registered 260 technical grade pesticides and 585 pesticide formulations. The total chemical pesticide consumption was 57353 Unit in the year of 2014-2015.

The World Health Organization reports every year that there are 3million pesticide poisonings, most of them were OP related, and 200,000 deaths worldwide that are attributed to either self-poisoning or occupational exposure. Globally 4.6 million tons of pesticide are annually sprayed into the environment, out of which only 1% is effective to target plants and rest 99% is released in non-targeted ecosystem like soil, water bodies and atmosphere (Global pesticide pollution). A vast majority of the population in India is engaged in agriculture and is therefore exposed to the pesticides used in agriculture. The rampant use of these chemicals, under the adage, "if little is good, a lot more will be better" has played havoc with human and other life forms.

Pesticides reaching the soil after spraying may affect soil quality by disturbing soil organisms such as earthworms, microbes and natural enemies which act as decomposers. This may decreases soil microbial biomass by directly killing or influencing activities such as behavior, reproduction and

metabolism of organism which may permanently impair and alter the closely interactive ecosystem (Gill and Garg, 2014). Pesticide residues in soils have the potential to cause toxicity to plants, their products and contaminate the food chain when taken up by plants roots. The accumulation of chlorpyrifos in soil has many adverse effects as at higher concentrations (10–300 mg kg^{-1}) it results in lowering the number of di-nitrogen fixing bacteria as well as total bacterial population. This leads to decrease in nitrogen and phosphorus content of soil (Down to Earth, 2001). In addition, the pesticide residues in soil and the environment have the tendency to pollute ground and surface water through leaching and surface run off, thereby increasing the risk of environmental contamination. Increased accumulation of pesticide residues in the food chain and drinking water have been reported to pose serious health hazards.

Contamination of water by pesticides is widespread. Pesticides can reach surface water through runoff from treated plants and soil followed by groundwater pollution. According to the USGS, at least 143 different pesticides and 21 transformation products have been found in ground water, including pesticides from every major chemical class. Increased accumulation of pesticide residues in the food chain and water have been reported to pose serious human health hazards (Akan *et al.,* 2013; Agbeve *et al.,* 2014). For instance, exposure to pesticide residues through food and water are reported to affect thyroid function, cause low sperm count in males, birth defects, increase in testicular cancer, reproductive (Mesnage *et al.* 2010) and immune malfunction (Tanner *et al.,* 2011), endocrine disruptions, cancers, immunotoxicity (Cocco *et al.,* 2013), neuro behavioural and developmental disorders (Gill and Garg, 2014).

This study therefore aims at assessing the levels of organophosphorus pesticide residues in soils, water and vegetable from some selected vegetable growing villages of Ottanchathiram, Dindigul District of Tamil Nadu.

MATERIALS AND METHODS

Study area

The study was taken in the Oddanchatram village, It is a town in Dindigul district in the Indian state of Tamil Nadu. Oddanchatram is a region in the southwest of Tamil Nadu. Oddanchatram is also famous for vegetable and cattle market. It is located at the base of the Western Ghats in South India. It is known as vegetable city of Tamil Nadu. Oddanchatram vegetable market is the largest supplying of vegetables in Tamilnadu and Kerala. Agriculture is the major economic support for the town region.

Sampling design

Eight intensive vegetable growing villages, Thasaraipatti, Veeralapatti, Ambilikai, Arasappapillaipatti, Vadakadu, Kosavapatti, Krishnakoundanputhur and Virupachi were selected for the pesticide residue analysis. Soil samples were collected randomly at depth 0–20 cm with a soil auger and put together to form a composite sample. The composite soil samples were well mixed and three soil replicates were collected from each vegetable growing villages. These gave a total of 72 soil samples from the study area. All soil samples were kept in well-labelled sampling bags and transported to the laboratory for analysis. The soil samples were air dried and sieved using 2 mm nylon mesh and sieved soils were taken for pesticide residues analysis.

Water sampling and charecterisation

Water samples were collected from eight villages in bore wells sources located within and/or around villages of intensive vegetable growing area (Chilli, Cauliflower, Brinjal and Bhendi) selected for the study. Wells were selected based on distance to vegetable growing field; 0–15 m and 16–30 m. Three replicates were collected from each well making a total of one hundred forty four (144) water samples. A water sampler was used to collect water samples into 1.5 L and 500 ml pre-cleaned polyethylene sample bottles with caps for pesticide residues and physico-chemical analysis respectively. The sampling bottles were rinsed with well water before taking the water samples. The samples were labelled and transported to the laboratory within 24–48 h on ice in clean ice chests and stored in the refrigerator at 4 °C until they were analysed pesticide residues. The samples for pesticide residues analysis were extracted within 24 h of arrival at the laboratory.

Extraction of pesticide residues from soil samples

Ten gram (10 g) of the representative soil samples were weighed and quantitatively transferred into 250 ml separating flasks. A 10 ml of acetonitrile was added to each of the soil samples in the flasks and ultra-sonicated for 5 min. An additional 10 ml of

acetonitrile was added, and the flasks closed tightly. The samples were placed on a horizontal mechanical shaker and set to shake continuous for 30 min at 300 rpm. The contents were then allowed to stand for 10 min to sufficiently separate the phases or layers. A 10 ml of the supernatants were carefully taken by pipette and dried over 2 g anhydrous magnesium sulphate through filter paper into 50 ml round bottom flasks. The concentrates were then adjusted to about 2 ml using the rotary film evaporator at 35 °C, and made ready for silica clean up step.

Clean up procedure for soil samples

Extracts clean up were done, using polypropylene. cartridge columns, packed with one-gram silica gel previously activated for 10 h in an oven at 130 °C, which have 1 cm thickness layer of anhydrous magnesium sulphate on top and conditioned with 6 mL acetonitrile. The concentrated extracts were then loaded onto the columns/ cartridges, and 50 ml pear shape flasks placed under the columns to collect the eluates. A 10 ml acetonitrile was used to elute the columns/cartridges afterwards. The total filtrates (eluents) collected were concentrated to dryness using the rotary evaporator set at 40 °C. The residues were re-dissolved in 1 ml ethyl acetate by pipetting and transferred into 2 ml standard opening vials prior to quantitation by gas chromatography (GC).

Extraction of pesticide residues from water samples

Water samples were filtered through 0.45 mL fiber glass filters to remove debris and suspended material, 1000 ml portions of the filtered water samples were transferred into 2 l capacity separating flasks. A 30 ml of saturated sodium chloride solution (NaCl) was added to each to produce a salt out effect in order to adjust the pH from 3.5 to 4. The samples were then thoroughly mixed by inverting the flask three to four times.

A 100 ml of dichloromethane as extraction solvent was then added to each sample and vigorously shaken manually for 2–3 min, while releasing the pressure intermittently. The phases were allowed to separate for 5 min and the dichloromethane extracts were separated from the aqueous layers. The extraction for each water sample was repeated twice with 100 ml of dichloromethane and the organic layers put together and dried over anhydrous sodium sulphate through filter papers into 50 ml round bottom flasks. The extracts from the water samples were then concentrated on rotary vacuum evaporators to about 1 ml and subjected to silica clean up.

Clean up procedure for water samples

Extracts clean up were done, using polypropylene cartridge columns, packed with one-gram silica gel previously activated for 10 h in an oven at 130 °C, which have 2 g layer of anhydrous sodium sulphate on top and conditioned with 6 ml dichloromethane. The concentrated extracts were then loaded onto the cartridges, and 100 ml round bottom flasks were placed under the columns to collect the eluates. A 20 mL dichloromethane was then used to elute the columns/cartridges afterwards, and the total filtrates (eluents) collected were concentrated just to dryness using the rotary evaporator set at 40 °C. The residues were re-dissolved in 1 ml ethyl acetate by pipetting and transferred into 2 ml standard opening vials prior to quantitation by gas chromatography (GC)

GC-MS Analysis

The Gas Chromatography - Mass Spectrometer from Thermo fisher, Trace-1300 series, were engaged for analysis. The instrument was set as follows, Injector port temperature set to 220° C, Interface temperature set as 250 °C, source kept at 220 °C. The oven temperature programmed as available, 75 °C for 2 mins, 150 °C at 10 °C min^{-1}, up to 250 °C at 10 °C min^{-1}. Split ratio set as 1:12 and the injector used was splitless mode. The DB-5 MS capillary standard non - polar column was used whose dimensions were 0.25mm OD x 0.25 µm ID x 30 meters length procured from Agilent Co., USA. Helium was used as the carrier gas at 1.5 ml min^{-1}. The MS was set to scan from 50 to 550 of ion source. The source was maintained at 220 °C and $4.5e^{-6}$ mtorr vacuum pressure. The ionization energy was -70eV. The MS was also having inbuilt pre- filter which reduced the neutral particles. The data system has inbuilt libraries for searching and matching the spectrum.

RESULTS AND DISCUSSION

Survey on use of pesticide and consumption pattern

The study was undertaken to evaluate the pesticide-use in vegetable cultivation in the Oddanchatram block of Dindigul district of Tamil Nadu. Choice of this area was deliberate because the Dindigul district constituted approximately eight per cent of the total vegetable area in the state. Chilli, Cauliflower,

Brinjal and Bhendi were the four vegetables considered for the study. These four crops are frequently sprayed with pesticides due to higher pest incidence. The area under Chilli, Cauliflower, Brinjal and Bhendi is 170,139,148 and 683 ha's in Ottanchathiram Block (Fig. 1) . Most of the farmers are using insecticides to control pests when compared to botanicals and biopesticides (Fig. 2)

Frequency of pesticide application

The study revealed that about 55 per cent the farmers applied 4 kg or less of technical grade pesticides per ha and the remaining 45 per cent used more than 4 kg of active ingredient (a.i)/ha in chillies and About 87 per cent of cauliflower growers applied pesticides amounting to 4 kg or less of a.i/ ha and the remaining applied more than this quantity. Average pesticide usage around 5, 2, 4 and 3 kg active ingredient per hectare on chillies, cauliflower, brinjal and bhendi crops, respectively. On an average, cauliflower and brinjal are each given 15 applications, chillies is given 13and bhendi is given 12 applications (Table 1 and 2)

Physico-chemical charecteristics of soil samples

The soil samples were collected from different vegetable growing field in eight villages and analysed for pH, EC(dSm^{-1}) and Organic carbon (%) (Table 3). The observed pH was in the ranges of 7.16 to 8.56 and highest pH was recorded in the village Arasappapillaipatti Cauliflower field soil sample (8.56) and lowest in the village Veeralapatti brinjal field (7.16). Most of the samples are showed pH above 7.5 and alkaline. The maximum EC (0.55 dSm^{-1}) was observed in the Bhendi field of Ambilikkai village and lowest (0.11 dSm^{-1}) in the village Thasarapatti Chilli field soil samples. The lowest organic carbon (0.11%) recorded in the bhendi field of Krishnagoundanputhur and higest (0.97%) in brinjal field of Arasappapillaipatti.

Chlorpyriphos and Monocrotophos residues in soil samples of Oddanchatram Block

The villages, Thasaraipatti, Veeralapatti, Ambilikai, Arasappapillaipatti, Vadakadu, Kosavapatti, Krishnakoundanputhur and Virupachi are intensive vegetable growing areas selected for the pesticide residue analysis. A total of 72 Soil samples were collected from these villages and analysed for chlorpyriphos and monocrotophos pesticide residues (Table 4) using GC-MS. The recovery range between 80-91% showed maximum extraction efficiency and analytical methods are correect for extraction of organo phosporous. Chloripyriphos residues occurred in all th soil samples analysed, ranged from 2.8 to 5.6 µg/kg. The maximum concentration in the Cauliflower (5.6 µg/kg) field of Thsarapatti village and minimun was in the Chilli

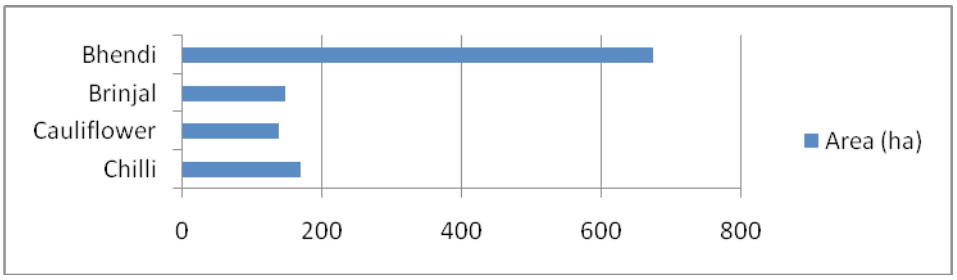

Fig. 1. Area under different vegetables in Oddanchatram Block

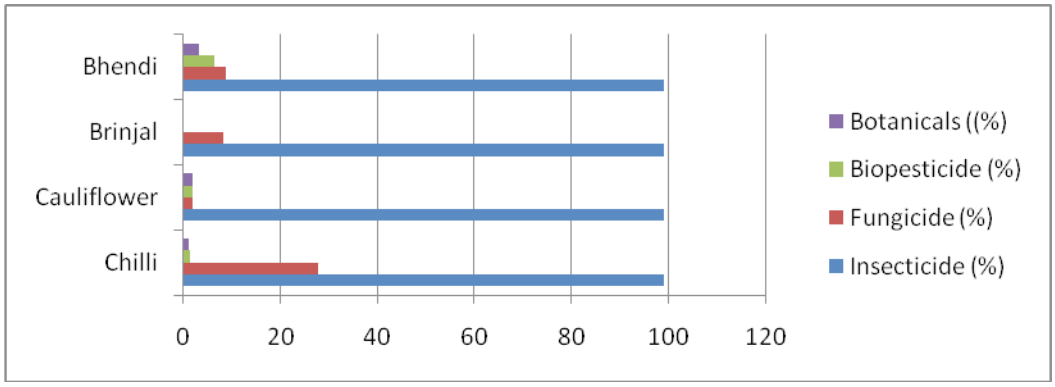

Fig. 2. Percentage of farmers using various type of plant protection chemicals

Table 1. Frequency of pesticide application

No. of Applications	Chilli (% of farmers)	Cauliflower (% of farmers)	Brinjal (% of farmers)	Bhendi (% of farmers)
< 11	4.40	0.0	0.0	20.0
11	5.97	4.1	0.0	26.0
12	20.1	11.0	5.71	20.0
13	28.2	18.0	8.57	13.0
14	23.8	20.0	20.0	10.0
15	5.6	24.0	22.8	6.6
>15	10.4	22.0	42.8	5.3

Table 2. Frequency of distribution and pesticide use intensity

Pesticides use intensity kg a.i/ha	Chilli (% of farmers)	Cauliflower (% of farmers)	Brinjal (% of farmers)	Bhendi (% of farmers)
< 2	5.9	24.0	—	3.3
2 – 3	10.3	44.0	—	36.0
3 – 4	38.1	18.0	31.4	23.0
4 – 5	23.1	8.9	31.4	20.0
5 – 6	11.9	—	31.2	10.0
>6	8.36	4.1	5.6	6.1

field (2.8 µg/kg). There is no significant variation of concentrations were observed among the villages, almost all the soil samples recorde around 3.5 to 4.8 µg/kg. The mean concentration observed among the different sites ranged from 3.8 to 5.6 µg/kg.

When compared to chlorpyriphos, the monocrotophos concentration was minimum in all the soil samples analysed. The concentration of monocrotophos renged from 1.5 to 4.2 µg/kg and maximum concentration (4.2) was found in Cauliflower field of Thasarapatti village and minimum was in Brinjali field of Veeralapatti (1.8). Overall, observation showed the pesticide usage was maximum in Cauliflower field, when compared to other crops. There is no significant variations observed among the concentration in different villges (Table 4). There wont be any correlation observed between pH and EC of the soils and pesticide residues but the organic carbon recored in Cauliflower field of Thasarapatti village showed maximum chlorpyriphos. The reason may due to absorption pf residues was maximum in high organic carbon soil samples.

The concentrations of chloepyriphos and monocrotophos residues in the soil samples suggests that vegetable growing farmers in the Ottanchathiram block use organophosphate pesticides in significant quantities

Chlorpyriphos residues in waterl samples of Oddanchatram Block

The residues of chlorpyriphos detected in the water samples collected at distances of 0-15m and 16 to 30m were summarised in the Tables 5 and 6. The maximum concentration of chloepyriphos recorded at a distance 0-15m of Cauliflower field, Krishnakoundanputhur village (0.8) and minimum was 0.1 in Vadakadu and Kosavapatti water samples. The water samples collected from the distance 16-30 m detected Nd to 0.02. The chlorpyrifos residue levels recorded in this study were far lower than the range of not detected (ND)-1545.1 µg/l reported by Essumang *et al.* (2009) in watersamples from Lagoons in Ghana.

Monocrotophos residues in waterl samples of Oddanchatram Block

The residues of monocrotophos detected in the water samples collected at distances of 0-15m and 16 to 30m were summarised in the Tables 6. The monocrotophos residues were very low when compared to Chlorpyriphos, Monocrotophos ranged from 0.1 to 0.55 at a distance of 0-15m, but in the distance 16-30m, the concentration ranged from Nd to 0.01. Water samples collected at a distance of 16-30m from Chilli, Brinjal and Bhendi fields showed concentrations from Not detectable to 0.01. When compared to brinjal and chilli and Bhendi fields, the concentration of monocrotophos was high in Cauliflower field. This showed cauliflower growing farmers are using high amount of monocrotophs to control pest and diseases in vegetables growing

Table 3. Characterization of soil samples

Villages	Vegetables	pH	EC (dSm^{-1})	OC (%)
Thasaraipatt	Chilli	8.11	0.11	0.72
	Cauliflower	7.76	0.13	0.80
	Brinjal	7.90	0.23	0.62
	Bhendi	7.46	0.24	0.82
Veeralapatti	Chilli	8.20	0.13	0.85
	Cauliflower	8.03	0.18	0.35
	Brinjal	7.16	0.37	0.40
	Bhendi	7.75	0.62	0.70
Ambilikai	Chilli	8.11	0.25	0.92
	Cauliflower	7.70	0.12	0.88
	Brinjal	8.43	0.23	0.88
	Bhendi	8.28	0.55	0.86
Arasappapillaipatti	Chilli	8.44	0.15	0.76
	Cauliflower	8.56	0.17	0.50
	Brinjal	8.26	0.15	0.97
	Bhendi	7.67	0.14	0.90
Vadakadu	Chilli	7.80	0.16	0.90
	Cauliflower	7.43	0.25	0.70
	Brinjal	7.41	0.30	0.37
	Bhendi	8.27	0.12	0.96
Kosavapatti	Chilli	7.88	0.47	0.40
	Cauliflower	7.28	0.42	0.43
	Brinjal	8.06	0.47	0.21
	Bhendi	8.34	0.35	0.32
Krishnakoundanputhur	Chilli	8.52	0.23	0.19
	Cauliflower	8.08	0.35	0.38
	Brinjal	8.46	0.55	0.44
	Bhendi	8.37	0.20	0.11
Virupachi	Chilli	8.07	0.15	0.11
	Cauliflower	8.17	0.23	0.80
	Brinjal	7.67	0.14	0.90
	Bhendi	7.80	0.16	0.29

Table 4. Average Concentration of Chlorpyriphos and Monocrotophos in soils of Ottanchathiram Block

Villages	Chlorpyriphos (µg/kg)				Monocrotophos (µg/kg)			
	Chilli	Cauliflower	Brinjal	Bhend	Chilli	Cauliflower	Brinjal	Bhendi
Thasaraipatt	2.8	5.6	4.5	4.0	2.3	4.2	4.0	3.3
Veeralapatti	3.5	4.6	4.3	3.2	1.8	2.8	1.5	1.9
Ambilikai	3.5	3.8	3.7	3.0	3.2	2.5	3.7	3.5
Arasappapillaipatti	4.3	4.8	4.6	4.1	3.2	3.8	3.3	3.3
Vadakadu	3.1	4.2	4.2	3.7	2.0	3.2	2.0	3.7
Kosavapatti	3.3	4.8	4.2	4.6	2.1	3.5	3.7	2.8
Krishnakoundanputhur	3.5	4.5	4.1	4.5	3.0	3.5	3.2	3.7
Virupachi	3.4	4.8	4.3	4.1	3.1	3.9	3.0	3.8

area. At a distance from 16-30m showed almost nil residues,because the transport of residues above 15m is less when compared to 0-15m.

CONCLUSION

The result from this studty revealed that the level of chlorpyriphos and monocrotophos contamination in soils and water samples of intensive vegetable growing area of Ottanchathiram block Chlorpyriphos is the most detected pesticide , when compared to monocrotophos. Among the vegetables, the usage of pesticide residues is maximum in Cauliflower field and observed contaminatio n level also maximum. The contamination of chlorpyriphos and monocrotophos

Table 5. Average Concentration of Chlorpyriphos in water samples of Ottanchathiram Block

Villages	Chlorpyriphos (µg/l)		Distance (0-15m)		Chlorpyriphos (µg/l)		Distance (16-30m)	
	Chilli	Cauliflower	Brinjal	Bhend	Chilli	Cauliflower	Brinjal	Bhendi
Thasaraipatt	0.20	0.50	0.40	0.20	0.1	0.2	0.1	Nd
Veeralapatti	0.10	0.30	0.50	0.10	Nd	0.01	Nd	0.05
Ambilikai	0.10	0.40	0.30	0.10	Nd	0.10	0.10	0.05
Arasappapillaipatti	0.25	0.45	0.25	0.20	0.10	0.10	0.03	0.10
Vadakadu	0.22	0.55	0.30	0.10	Nd	0.02	Nd	Nd
Kosavapatti	0.35	0.50	0.32	0.10	0.10	0.10	0.10	Nd
Krishnakoundanputhur	0.35	0.80	0.40	0.30	0.10	0.10	0.20	Nd
Virupachi	0.20	0.40	0.30	0.30	Nd	Nd	Nd	Nd

Table 6. Average Concentration of Monocrotophos in water samples of Ottanchathiram Block

Villages	Monocrotophos (µg/l)		Distance (0-15m)		Monocrotophos (µg/l)		Distance (16-30m)	
	Chilli	Cauliflower	Brinjal	Bhend	Chilli	Cauliflower	Brinjal	Bhendi
Thasaraipatt	0.20	0.30	0.25	0.10	0.01	0.02	0.01	Nd
Veeralapatti	0.10	0.50	0.30	0.20	0.01	0.01	Nd	Nd
Ambilikai	0.10	0.73	0.30	0.25	Nd	0.02	Nd	Nd
Arasappapillaipatti	0.20	0.40	0.55	0.20	Nd	0.01	0.1	0.01
Vadakadu	0.10	0.30	0.45	0.10	Nd	Nd	Nd	Nd
Kosavapatti	0.10	0.40	0.25	0.10	Nd	Nd	Nd	Nd
Krishnakoundanputhur	0.10	0.30	0.55	0.10	Nd	0.01	Nd	0.01
Virupachi	0.40	0.20	0.40	0.20	0.10	0.01	Nd	Nd

in soil and water samples pose a serious threat to environment and human health. There may be chances of leaching to nearby water bodies and ground water. The residues of chlorpyriphos and monocrotophos decresed at 16-30 m of distances when compare to 0-15 m. The chlorpyriphos and monocrotophos residues at 16-30 m distance shows, the residues not pose health risk to humanbeing, but still intensive study is necessary for the distance above 30m. Continuous monitoring of pesticide residues is necessary in the study area, because these villges are intensive vegetable growing area using pesticides to control pest and diseases.

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