EPH - International Journal of Biological & Pharmaceutical Science
ISSN (Online): 2208-2166
Volume 02 Issue 01-August -2016

DOI: https://doi.org/10.53555/eijbps.v2i1.14

MEASUREMENT OF CHEMICAL PESTICIDE RESIDUES IN CAPSICUM GROWN IN SOUTH INDIA

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Abstract:-

Capsicum (also known as peppers) is a genus of flowering plants in the nightshade family Solanaceae.Capsicum grown in five districts of Karnataka (Bangalore urban, Bangalore rural, Kolar, Chikkaballapura and Ramanagara) were analyzed for 20 pesticide residues by Gas liquid Chromatography equipped with ECD and FCD. Recovery studies performed at 0.1, 0.5 and 1.0mg kg^{-1} fortification levels of each compound and recoveries obtained ranged from 743%-97% with relative standard deviation lower than 7.5%. The method showed the good linearity over the range assessed 0.01-1.0mg kg^{-1} respectively. Capsicum was analyzed for acephate, chlorphyriphos, dichlorvos, phorate, deltamethrin, fenvalerate and cyfluthrin-ù residues. Except phorate none of the residues in capsicum crossed the MRL. Phorate was detected in samples from Ramanagara district while 12.5% samples from Bangalore rural, chikkaballapura and Kolar district showed phorate residues exceeding the MRL value of 0.05mg kg^{-1}. Cyfluthrin-ù residue is in higher proportion in capsicum samples from Bangalore rural than Bangalore urban. It is therefore required to investigate extensively the monitoring studies covering all the vegetables crops from different agro-climatic zones of Karnataka to know the exact status of pesticide contamination.

Keywords:- *Pesticide, capsicum, monitoring, MRL*

INTRODUCTION:

Capsicum (also known as peppers) is a genus of flowering plants in the nightshade family Solanaceae. Its species are native to the Americas, where they have been cultivated for thousands of years. Following the Columbian Exchange, it has become cultivated worldwide, and it has also become a key element in many cuisines. In addition to use as spices and food vegetables, *Capsicum* species have also been used as medicines and lachrymatory agents.

India enjoys salubrious climatic condition throughout the year and vegetables and fruits are grown abundantly to suffice the diet of human beings. Consumption of vegetables necessitates the nutrient requirements of the body to protect the body against the incurable diseases and to provide immunity. Vegetables grown in nature are prone to infested by pest and pathogen and becomes inevitable on the part of farmers to save the crop from any eventualities which may found to thwart the yield and quality of the crop.

A chemical in the form of pesticide is used as a precautionary measure to eliminate pest and pathogens which may damage the crop and sometimes to control the infestations. The sprayed pesticide may vaporize due to atmospheric pressure and temperature, photolysis, Microbial metabolism and permeating inside the plant body which causes deleterious effect on the lives of human beings and domestic animals. Vegetables with pesticide residue when consumed remains on the adipose tissue without being degraded or destroyed. . The problem is especially serious when these commodities are consumed (Solecki et al.,2005) and found to damage the vital organs of the body (Berrada et al., 2010).Insecticide use in India is alarmingly high (75%)compared to 32% in the world. It is estimated that 20% of contamination is above MRL and is causing lot of havoc amongst the Indian population. The health benefits of capsicum include relief from cancer, peptic ulcer, menopausal problems, low risk of cardiovascular disease and diabetes. It has anti-inflammatory, analgesic properties and may also provide relief in pain related to arthritis. It also provides relief from fibromyalgia, skin aging and psoriasis.

Materials and methods

Field study was conducted to collect the information about various aspects of pesticide use and their safety. This information was used as the baseline data to investigate the residue level of the analyzed pesticides in vegetables. The present investigation study was conducted during the year 2014-2015 in five districts of Karnataka.

Study area

Five districts of Karnataka namely, Bangalore rural, Bangalore urban, Chikkaballapura, Kolar and Ramanagara are presenting the hub of agricultural activities were selected as study areas for the determination of pesticide residues in capsicum samples. The Karnataka covers an area of 191,976 square kilometers (74,122 sq mi) or 5.83% of the total geographical area of India

Sampling procedure transportation and storage

Randomly selected vegetable sample capsicum collected from the growing areas and district headquarters of five districts (Including major markets of Bangalore urban). Samples of marketable size (1kg each) were collected and transported to the laboratory in fresh plastic bags and kept in refrigerator (5°C) until analysis in order to avoid any Degradation of residues between sampling and analysis. Only the edible parts of vegetables Were processed for residue analysis. A total of 320 vegetables samples (viz., 8 samples x 8 different vegetables x 5 districts) were collected from five different districts of Karnataka.

Standard solutions

Pesticide standard stock solutions were procured from Indian Agricultural Research Institute (IARI), New Delhi. Working standard solutions containing a mixture of the analyte were prepared from the stock by appropriate solvent dilutions in n-hexane.

Pesticide analysis
Preparation of the samples and determination of insecticide residues was based on the method described by AOAC (2000). During this investigation, residues of insecticides in vegetables (beans, brinjal, cabbage, carrot, cauliflower, chilly, lady's finger and tomato) were monitored in γβ0 vegetable samples from five districts of Karnataka using Gas Chromatograph with ECD and FTD (Shimadzu make, Model GC-2010).

Extraction
The extract was then passed through a layer of sodium sulfate (5g) and evaporated to dryness in a rotary evaporator at a temperature below 40.

In the present study, only the edible parts of vegetable samples (1.0kg) were chopped and 50g of samples were extracted in a warring blender with 100 ml acetonitrile for 2-3min. The solvent was filtered through a Buchner funnel. The fruit residue was again subjected to extraction with 50ml acetonitrile two more times. The extracts were evaporated under vacuum to about 5ml and then transferred to a separator funnel of capacity 1000 ml. 600 ml of 5% sodium chloride was added and the extract was exchanged into petroleum ether layer by liquid-liquid partitioning thrice (100ml, 2 × 50ml).

Clean up
The glass beaker containing extract was rinsed with acetone and was transferred to the column, which was allowed to stand for 45min. Subsequently, the petroleum ether present in the column was eluted drop-wise (5ml/min). Glass column (60cm length × 2.0cm I.D) was packed with a mixture of florisil (10g), anhydrous sodiumsulphate (10g) and activated charcoal (0.2g) supported on a cotton plug was used for cleanup and the sample was wetted with 50ml petroleum ether. Sample slurry prepared using petroleum etherwas transferred to the column. When about 5ml petroleum ether remained on the surface of the adsorbent, the extract was eluted with 200ml each of freshly prepared 6% solvent mixture (diethyl ether in petroleum ether), 15% solvent mixture (diethyl ether in petroleum ether) and 50% solvent mixture successively. The eluents were concentrated to dryness in a rotary evaporator under vacuum and diluted to 10ml with n-hexane for further analysis. From the dissolved residues, 1µl was injected to gas chromatograph and peak areas were compared with those obtained from similar injections of standards.

Pesticide residue analysis
Insecticides like organochlorines (OCs) and pyrethroids (SPs) were analyzed using ECD (63Ni) and a capillary column BP-5 (60m × 0.25mm I.D. × 0.25µm film thickness) with split ratio 1:10. Nitrogen flow rate of 30ml/min, injection port temperature of 250^0C and temperature of detector of 300^0C and an injection volume of 1µl were the Gas - Liquid Chromatography (GLC) working conditions maintained during the analyses. The pesticide residue analysis was performed on Gas Chromatograph GC-2010 (Shimadzu make) equipped with ECD (Electron Capture Detector) and FTD (Flame Thermionic Detector). A fused silica capillary column (BP5- 5% Phenyl, 95% Dimethylpolysiloxane) was used for the analysis. The column temperature was initially maintained at 80^0C for 5min and then slowly increased to 260^0C at the rate of 10^0C per min for 5min and finally increased to 290^0C for 5min. In contrast, organophosphates (OPs) residues were analyzed with FTD and a split less capillary column DB-1 (10m × 0.53mm I.D. × 2.65µm film thickness). The GLC working conditions maintained during the analyses were nitrogen flow rate of 60ml/min, hydrogen flow rate of 3ml/ min, air flow rate of 150ml/min, injection port temperature of 280^0C, detector temperature of 300^0C and an injection volume of 1µl with split ratio of 1:10. The column temperature was initially maintained at 180^0C for 5min and then gradually increased to 260^0C for 5min.

Estimation / Quantification of residues
The sample injector is maintained at a temperature higher than the boiling point of the highest boiling component of sample in order to ensure rapid vaporization of the liquid samples. The carrier gas entering the sample injector sweeps off the vaporized sample and passes down the thermostated or temperature programmed column. The carrier gas obtained from a steel gas cylinder passes through a flow regulator for the adjusted flow rate and enters into the sample injector. A little amount of the sample is introduced into the sample injector with the help of a hypodermic syringe. The components of the sample are distributed between the stationary and the mobile phases and pass down the column at different rates. This results in the separation of the components of the sample. The carrier gas with the separated components enters the detector, which measure the change in composition of the carrier gas as it passes through it. This change is amplified before it is fed into a recorder which drives the recording pen on a moving strip of paper and a chromatogram is obtained. Currently rapid instrumental methods are available for data processing and obtaining chromatograms in computer compatible formats.

The pesticide residue concentration was calculated using the equation,

$$\text{Residues value } (\mu g/g) = \frac{A_s \times V_{std} \times C_{std} \times D_f}{A_{std} \times V_s \times W_s}$$

Where
A_s = peak area of sample injected (mv)
A_{std} = peak area of standard injected (mv)
V_s = volume of sample injected (µl/ml)
V_{std} = volume of standard injected (µl/ml)

C_{std} = concentration of standard (µ g/ml)
W_s = weight of sample taken (g)
D_f = dilution factor (ml)

Efficiency of the method was validated with recovery.Among the quantification of the targeted 20 residues,nine are organochlorines (viz., aldrin, dieldrin, endosulfan-α, endosulfan-ù, endosulfansulphate, HCH-α, HCH-ù, HCH-Û, heptachlor), six are organophosphorus (i.e., acephate, chlorpyriphos, dichlorvos (DDVP), monocrotophos, phorate, profenophos) and five are synthetic pyrethroids (cyfluthrin-ù, cyhalothrin-Ʒ, cypermethrin, deltamethrin, fenvalerate). By injecting 1µl of the standard solution or the cleaned up extract into the GC, retention times (RT) and peak areas of analytes were recorded. Residues were estimated by comparison of peak heights/peak areas of the standards with that of the unknown or spiked samples run under similar conditions.

Fortification / Recovery studies
Spiked samples were calculated in the same way as regular samples (Harry *et al*., 1993; Michel *et al*., 2003; Anna *et al*., 2004).The recovery studies for 3 replicates for each pesticide at three different fortification levels (1.0, 0.5 and 0.01mg/kg) were carried out. For this purpose, vegetable samples were spiked with 1ml of desired concentration of pesticide. Resulting samples were mixed and allowed to stand for 30 min before extraction and then processed separately as per the methodology described above. The amount of pesticide residues in vegetable samples were calculated by measuring peak areas from extracted current profiles and comparing with those obtained from matrix-matched standards of a concentration similar to that of samples.

Calculation of Percentage Recovery
The percentage recovery was calculated using the formula

$$\% \text{ Recovery } = \frac{\text{Amount recovered}}{\text{Amount spiked}} \times 100$$

Results and discussion:
Variation in acephate, chlorpyriphos, dichlorvos, monocrotophos, phorate, cyfluthrin-ù, cyhalothrin-Ʒ, cypermethrin, deltamethrin and fenvalerate residues in capsicum samples are detailed below (Table-1).

Acephate residues
These results are also in fair agreement with our findings.It varied from 0.171 to 0.333mg/kg (mean = 0.127mg/kg), 0.015 to 0.032mg/kg (mean = 0.042mg/kg), 0.172 to 0.325mg/kg (mean = 0.092 mg/kg), 0.172 to 0.285 mg/kg (mean = 0.086mg/kg) and 0.024 to 0.324mg/kg (mean = 0.044mg/kg) in the capsicum samples collected from Bangalore rural, Bangalore urban, Chikkaballapura, Kolar and Ramanagara respectively.

Acephate is detected in 37.5% of samples collected from Chikkaballapura and Kolar districts, 25% of the samples from Ramanagara and Bangalore urban, and 50% of samples from Bangalore rural districts, but none of samples exceeded the MRL of 2.0mg/kg. The trend of mean concentration of acephate residue in capsicum in different districts is Bangalore rural > Chikkaballapura > Kolar > Ramanagara > Bangalore urban. Crentsil Kofi Bempah *et al.,* (2012) studied chlorpyrifos residues levels in 309 samples of fruits and vegetables (pineapple, lettuce, cabbage, cucumber and onion) samples sold in Ghanaian markets, with the residue level varying from 0.001-0.062 mg/kg. They also found that chlorpyriphos in pineapple was higher than their respective European Commission MRLs. Mean concentration of chlorpyrifos was reported in eggplant (β4.0β3g/kg), cabbage (10.553g/kg), cauliflower (β.853g/kg), tomato (178.87 3g/kg) and ladyfinger (β.4λ3g/kg) from Hyderabad, Andhra Pradesh, India (Sukesh *et al.,* 2012).

Chlorpyriphos residues
Chlorpyriphos residue in Capsicum samples are in the range of 0.112 to 0.151mg/kg (mean = 0.048mg/kg) with 37.5% samples contamination in Bangalore rural, 0.014 to 0.015mg/kg (mean = 0.004mg/kg) with 25% of sample contamination in Bangalore urban, 0.112 to 0.153mg/kg (mean = 0.033mg/kg) with 25% sample contamination in Chikkaballapura and 0.115 to 0.152mg/kg (mean = 0.033mg/kg) with 25% of samples contamination in Ramanagara district. chlorpyriphos was not detected in samples from Kolar district. None of samples showed chlorpyriphos residue above the MRL value of 0.2mg/kg. The trend of mean concentration of chlorpyriphos residue in capsicum in different districts is Bangalore rural > Chikkaballapura > Ramanagara > Bangalore urban > Kolar.

Dichlorvos residues
Dichlorvos residue was not detected in capsicum samples from Bangalore urban, Chikkaballapura and Ramanagara districts. In the samples from Bangalore rural and Kolar districts, it varied from 0.022 to 0.041mg/kg (mean = 0.008mg/kg) with 25% of samples contamination and 0.042mg/kg (mean = 0.005mg/kg) with 12.5% of samples contamination respectively in Bangalore rural and Kolar districts. None of samples exceeded the MRL of 0.15mg/kg. The trend of mean concentration of dichlorvos residue in capsicum in different districts was Bangalore rural > Kolar.
Beena Kumari *et al.,* (2003) who reported dichlorvos residue concentration ranging from 0.004 – 0.022mg/kg in cabbage, cauliflower, pea grains, brinjal, tomato, potato and green chilly samples collected from wholesale markets of Hisar, Haryana.

Phorate residues

In the capsicum samples collected from Bangalore rural, Bangalore urban, Chikkaballapura and Kolar districts, the phorate residue concentration was found to range from 0.039 to 0.064mg/kg (mean = 0.013mg/kg), 0.029 to 0.043mg/kg (mean = 0.014mg/kg), 0.037 to 0.068mg/kg (mean = 0.068mg/kg) and 0.037 to 0.064mg/kg (mean = 0.023mg/kg) respectively. The sample contamination accounted for 25% for Bangalore rural and 37.5% each for Bangalore urban, Chikkaballapura and Kolar districts. Phorate residue was not detected in samples from Ramanagara district while 12.5% of samples from Bangalore rural, Chikkaballapura and Kolar districts showed phorate residue exceeding the MRL value of 0.05mg/kg. The trend of mean concentration of phorate residue in capsicum in different districts is Kolar > Chikkaballapura > Bangalore urban > Bangalore rural > 6 Ramanagara. Ligang wang *et al.,* (2008) who revealed the presence of phorate in Shanghai green (0.0257µ g/g) and Chinese cabbage (0.0398µg/g) from Nanjing, China. The present investigation results are also endorsed by findings of Chen et *al.,* (2011) who reported phorate residues in fruits and vegetables which vary from BDL to 0.405mg/kg from Xiamen, China.

Cyfluthrin-β residues

The concentration of cyfluthrin-ù residue in capsicum samples from Bangalore rural and Bangalore urban ranged from 0.031 to 0.045mg/kg (mean = 0.01mg/kg) and 0.012 to 0.042mg/kg (mean = 0.007 mg/kg), each district with 25% of sample contamination. None of samples were having residue values above the MRL of 3.0mg/kg. Cyfluthrin-ù residue was not detected in samples from Chikkaballapura, Kolar and Ramanagara districts. Cyfluthrin-ù residue is in higher proportion in capsicum samples from Bangalore rural than Bangalore urban.

Deltamethrin residues

The concentration of deltamethrin in capsicum samples varied from 0.332 to 0.381mg/kg (mean = 0.089mg/kg) with 25% sample contamination, 0.251mg/kg (mean 0.0.031mg/kg) with 12.5% sample contamination, 0.333 to 0.381mg/kg (mean = 0.089mg/kg) with 25% sample contamination and 0.024 to 0.033mg/kg (mean = 0.007mg/kg) with 25% sample contamination respectively in Bangalore rural, Bangalore urban, Kolar and Ramanagara districts. Deltamethrin residue was not detected in samples from Chikkaballapura district. None of the samples showed deltamethrin residue higher than the MRL of 0.2mg/kg. The trend of mean concentration of deltamethrin residue in capsicum in different districts was Bangalore rural > Kolar > Bangalore urban > Ramanagara > Chikkaballapura. The results of the present investigation are higher than the findings of a similar survey conducted by Beena Kumari and Kathpal (2009) who reported 68% contamination in vegetarian diet samples with deltamethrin residue (0.008 -0.102mg/kg), represented by samples from homes, hostels and hotels from Hisar, Haryana.

Fenvalerate Residues

In Bangalore rural, the concentration of fenvalerate residue in capsicum varied from 0.031 to 0.123mg/kg (mean = 0.019mg/kg) with 25% sample contamination. In Chikkaballapura, Kolar and Ramanagara districts, it ranged respectively from 0.121mg/kg (mean= 0.015mg/kg), 0.032mg/kg (mean = 0.004mg/kg), 0.111 mg/kg (mean = 0.014mg/kg), with each district having 12.5% sample contamination. Fenvalerate residue was not detected in Bangalore urban district samples. Fenvalerate residue in none of samples crossed the MRL value of 1.0mg/kg.The trend of mean concentration of fenvalerate residue in capsicum in different districts is Chikkaballapura > Ramanagara > Kolar. Maria *et al.,* (2011) who reported the presence of fenvalerate in the range of 0.061-0.07mg/kg in vegetable samples like green pepper, white onion, tomato, lettuce, green onion, potato and saladette tomato produced in Sonora, Mexico.

Monocrotophos, Cyhalothrin-λ and Cypermethrin residues

The monocrotophos, cyhalothrin-Ʒ and cypermethrin residues are below the detectable level in capsicum samples from all the five districts. Ranga Rao *et al.,* (2009) revealed the presence of monocrotophos in the range of 0.001-0.044mg/kg in vegetable samples (viz., brinjal, cucumber, okra, ridge gourd and tomato) collected from Andhra Pradesh, India. Crentsil Kofi Bempah *et al.,* (2011) who showed the residual levels of cypermethrin detected are in the range of 0.004-0.008mg/kg (mean: 0.004mg/kg) and 0.030-0.080mg/kg (mean: 0.060mg/kg) respectively for pear and lettuce samples collected from markets in Kumasi.

Recovery studies

Our findings are corroborated by the earlier works listed. Beena Kumari *et al.,* (2002) reported the per cent recoveries for OC, OP, SP and carbamate respectively varied from 80– 111, 83–125, 73–95 and 82–104% at spiking levels of OC (0.01–0.1ppm), SP (0.25ppm), OP (0.25–0.5ppm) and carbamates (0.5 –1ppm). Darinka and Lucija (2003) reported for 90 pesticides in fruits from Slovenia in the concentration range of 0.01 to 0.50mg/kg, with the pesticide recoveries greater than 80%. Doyeli *et al.,* (2011) reported mean recoveries of pesticides in spinach and eggplant in the range of 70-120% and RSD less than 10%. Gouri *et al.,* (2011) reported recoveries of pesticides in fruits and vegetables which ranged between 72 and 114% with a RSD < 20%.

In order to check the authenticity of the experimental procedure followed for extraction of different pesticides from capsicum samples, recovery studies were performed. All the targeted pesticides (aldrin, dieldrin, endosulfan-α endosulfan-ù, endosulfan sulphate, HCH- α, HCH-ù, HCH-Û, heptachlor, acephate, chlorpyriphos, dichlorvos(DDVP), monocrotophos, phorate, profenofos, cyfluthrin-ù, cyhalothrin-Ʒ, cypermethrin, delta methrin and fenvalerate) were spiked at 0.01, 0.5 and 1.0 mg/kg in capsicum.Percent recoveries of insecticides for the vegetable indicated that recoveries

are good and performance of extraction, clean-up and chromatographic parameters for the analysis of pesticide residues in vegetable samples are good. Recovery rate of 76.6 to 96.5% in capsicum was noticed.The variation in the recovery is not always possible to predict accurately but may be due to complete evaporation of the solvents on the rotary evaporator, oxidation due to solvent evaporation through direct air and degradation of pesticides wheeler et al., (1983).

Similar results also reported by Maria *et al.,* (2011) for vegetable samples (77–115%). Srivastava *et al.,* (2011) also investigated the recovery rates of different insecticides in vegetables which vary from 70.22 to 96.32%, with RSD of 15%. Xingang *et al.,* (2011) determined the average recoveries of insecticides in fruits and vegetables which ranged from 74.2 to 112.5%, with RSDs of 1.4-13.8%. Qozowicka *et al.,* (2012) investigated mean recoveries of pesticides for vegetables spiked at three fortification levels (0.001-2.5mg/kg) which ranged from 70.07 to 118.90% with RSDs ranging from 0.15 to 8.58.

Conclusion:

Capsicum samples contaminated with pesticide residues accounted for 35% acephate, 22.5% chlorpyriphos, 7.5% dichlorvos, 27.5% phorate, 17.5% delta-methrin and 12.5% fenvalerate..Majority of the samples collected from five districts showed mean concentration of acephate, chlorpyriphos, cyfluthrin-ù, cyhalothrin-Ʒ, cypermethrin, deltamethrin, dichlorvos, fenvalerate, monocrotophos and phorate residues below their respective MRL values. The results of the survey indicated that vast majority of growers use pesticides non judicously and indiscriminately ignoring pre harvest period. This is a severe socio-political concern to be tackled holistically and the farmers should be made more alert of ill-effects of excess use of pesticides. Regular consumption of contaminated vegetables could pose potential health hazards.

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Table-1 showing Pesticide residues (mg/kg) in capsicum samples

Note: BDL= below detection limit, n= No. of samples analyzed, a= Contaminated, b= % of contamination

Sl. No.	Pesticides	Residue range (mg/kg)																			
		Bangalore rural (n=8)				Bangalore urban (n=8)				Chikkaballapura (n=8)				Kolar (n=8)				Ramanagara (n =8)			
		a(b)	min	max	mean	a(b)	min	max	mean	a(b)	min	max	mean	a(b)	min	max	mean	a(b)	min	max	mean
1	Acephate	4(50)	0.171	0.333	0.127	2(25)	0.015	0.032	0.042	3(37.5)	0.172	0.325	0.092	3(37.5)	0.172	0.285	0.086	2(25)	0.024	0.324	0.044
2	Chlorpyriphos	3(37.5)	0.112	0.151	0.048	2(25)	0.014	0.015	0.004	2(25)	0.112	0.153	0.033	BDL	BDL	BDL	BDL	2(25)	0.115	0.152	0.033
3	Dichlorvos (DDVP)	2(25)	0.022	0.041	0.008	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	1(12.5)	BDL	0.042	0.005	BDL	BDL	BDL	BDL
4	Monocrotophos	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
5	Phorate	2(25)	0.039	0.064	0.013	3(37.5)	0.029	0.043	0.014	3(37.5)	0.037	0.068	0.018	3(37.5)	0.037	0.064	0.023	BDL	BDL	BDL	BDL
6	Cyfluthrin-û	2(25)	0.031	0.045	0.01	2(25)	0.012	0.042	0.007	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
7	Cyhalothrin-ʒ	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
8	Cypermethrin	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
9	Deltamethrin	2(25)	0.332	0.381	0.089	1(12.5)	BDL	0.251	0.031	BDL	BDL	BDL	BDL	2(25)	0.333	0.381	0.089	2(25)	0.024	0.033	0.007
10	Fenvalerate	2(25)	0.031	0.123	0.019	BDL	BDL	BDL	BDL	1(12.5)	BDL	0.121	0.015	1(12.5)	BDL	0.032	0.004	1(12.5)	BDL	0.111	0.014