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Research article

Cypermethrin residue analysis of fruit and soil samples in eggplant ecosystem in Bangladesh

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Abstract
Cypermethrin insecticide is now indiscriminately used by the farmers of Bangladesh in eggplant (*Solanum melongena* L.) to protect from eggplant shoot and fruit borer (ESFB). The aim of this study was to determine the persistence of cypermethrin residues in eggplant fruit and in the soil of the eggplant crop field. Different concentrations of cypermethrin (1 ml/L and 2 ml/L) insecticide were applied in the eggplant field by the knapsack sprayer and the residues were analyzed by Gas Chromatograph-Electron Capture Detector technique. The results showed that cypermethrin residues determined from fruit and soil samples sprayed at the rate of 1 ml/L in the field were above maximum residue limits (MRL) up to three days after spraying (0.762 ppm) in fruit samples and up to five days after spraying in soil (0.608 ppm). In case of spraying 2 ml/L of cypermethrin, fruit samples have residues above MRLs up to five days after spraying (0.753 ppm) and soil samples have up to seven days after spraying (0.768 ppm). The results of this study suggested that the eggplant vegetable should be consumed at least three and five days after spraying cypermethrin if it was treated with 1 and 2 ml/L of cypermethrin, respectively.
Keywords: Cypermethrin, degradation, ecosystem, eggplant, residue.

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Introduction

Eggplant fruit is extensively damaged by the most destructive pest, shoot borer (ESFB) (*Leucinodes orbonalis*) which caused 31-86% fruit damage in Bangladesh that makes it totally unfit for human consumption [1]. To overcome this loss, use of pyrethroid insecticides like cypermethrin is commonly used by the farmers of Bangladesh because of its high efficiency [2] and is applied indiscriminately even at fruiting stage [3]. Indiscriminate and haphazard use of these chemicals, particularly at fruiting stage, leads to its accumulation in the vegetables, which consequently cause hazards to human beings through the food chain [4]. This results in serious contamination of different component of the environment (surface waters, aquifers, soil, air etc.) including human, wildlife and other organisms [5]. Due to lack of knowledge about pesticides, our farmers are severely affected by these toxic pesticides as well as toxify the environmental components. Residues of cypermethrin insecticide persist in soil and edible parts of the crops such as fruits [6]. So, there is demand to create interest among the people regarding the accumulation of residues of the toxic chemicals in daily food. Nevertheless, no study so far has been conducted about the residue of cypermethrin in eggplant in Bangladesh.

Therefore, the present study was undertaken to analyze the residue status of cypermethrin from fruits and soil samples and to find out that when vegetable consumption is safe for human beings after spraying cypermethrin.

Materials and methods

Fruits and soil sample collection

Different concentrations (1ml/L and 2ml/L) of cypermethrin (Ripcord 10 EC) in water were applied in the experimental eggplant field (each plot 3m × 3m) considering Randomized Complete Block Design (RCBD) with three replications. Representative fruit and soil samples were collected randomly at 0, 1, 3, 5, 7, 10, 12 and 15 days after spraying (DAS) for determining the residues present in the fruits and soil. The collected samples were kept in deep freeze (-22^{0}C). The residue analysis was done at Insecticide Toxicology Laboratory in the Department of Entomology, Bangabandhu Sheikh Mujibur Rahman Agricultural University, Gazipur. Gas Chromatograph (GC)-17A and Electron Capture Detector (ECD) was used to quantify the residue level in fruit and soil samples. The standard cypermethrin was obtained from Sigma-Aldrich Laborchemikalien, Seelze, Germany through Arnica Scientific Pvt. Ltd. Dhaka, Bangladesh. The formulated product of cypermethrin (Ripcord 10

EC) was tested in the laboratory and found to be 100% pure.

Extraction of cypermethrin from fruit samples

The frozen samples were taken from the deep freeze and kept at room temperature for 5-6 hours of thawing. The methodology prescribed by William and George [7] with necessary modifications was adopted for extraction, separation and cleanup of the sample. Around 250 g eggplant fruit sample was grounded thoroughly with the meat grinder (Hand Mixer M-122, Bamix, Switzerland). From this, 20 g sub sample was taken into a wide mouth jar (250 ml), and then 100 ml of hexane was added to it. Sodium sulfate (Na_2SO_4) was also added to the sample until the water was removed. The mixture was then macerated with mortar pestle. The homogenized material was then poured into 250 ml conical flask and placed on a shaker (Orbital Shaking Incubator) for 6 hours continuous shaking. After shaking, the slurry was filtered through a Buchner funnel with suction. The flask and filter cakes were rinsed with 8-10 ml of hexane each. The filtrate was then transferred into 250 ml round bottom flask and was dried to 5-7 ml by evaporation using a rotary vacuum evaporator. The concentrated filtrate was then transferred into 500 ml separatory funnel making 10 ml volume with hexane. For color removal, around 20 ml methanol was added with 10 ml filtrate and shaken vigorously for 3-5 minutes in a shaker. After shaking, the separatory funnel was set on a stand and kept undisturbed for 3-5 minutes. Then the clear part of the solution from the bottom of the separatory funnel was collected in a vial which was then centrifuged at 12000×g for 5 min. After centrifuging, the supernatant was cleaned up by SPE cartridge. Then the final volume was adjusted to 10 ml and 2 µl was used for injection.

Extraction of cypermethrin from soil samples

One kg soil sample was collected from the eggplant plot of which 100 g representative sample was taken. From this representative sample, 20 g of working sample was taken in a conical flask (250 ml) and then 100 ml of hexane was added to it. Sodium sulfate (Na_2SO_4) was also added to sample until the water was removed from it. The conical flasks were capped by aluminium foil and placed in the orbital shaking incubator for 6-12 hours with 120-140 rpm at a temperature of 20°C. The extracted solutions were then collected in the centrifuge tubes. The solutions were centrifuged for 10 minutes at 16500×g and 20°C. The solutions were filtered through Whatman paper and the final extract solutions were collected in the round bottom volumetric flask (250 ml). Then the solutions were dried by rotary vacuum evaporator until 5-7 ml extracts remained. The extracted solutions were collected in the centrifuge tubes and centrifuged for 10 min at 16500×g and 20°C. After centrifuging, the supernatant was cleaned up by SPE cartridge. Then the final volume was adjusted to 10 ml and 2 µl was used for injection.

Quantification of cypermethrin in samples

The concentrated extracts were subjected to analysis by GC-17A (Shimadzu) with Electron Capture Detector (ECD). The capillary column used was AT-1, length 30 m, ID 0.25 mm and film thickness 0.25 µm. Nitrogen was used as a carrier and make up gas in ECD. The instrument parameters for GC-ECD to quantify the cypermethrin residue were as follows: Injection mode split; temperature 280°C; flow control mode linear velocity and split ratio 10. The initial column temperature was 150°C, later increased to 160°C, held for 1 min and then increased to 270°C at the rate of 10°C/min and held for 6 min. The ECD temperature was 300 °C, current 1.00 pA and makeup flow 30 ml/min. Total run time was 18 min. Prior to injection of the sample extract, standard solutions of different concentrations of cypermethrin were prepared and injected with the above instrument parameters. The sample was calibrated (retention time, peak area etc.) against four pointed calibration curve of standard solution of concentrated insecticide. Each peak was characterized by its retention time. Sample results were expressed in ppm automatically by the GC software which represented the concentration of the final volume injected. From this value the actual amount of insecticide residue present in the sample was determined by using the following formula:

$$\frac{\text{Concentration in injection volume (ppm)} \times \text{Quantity of final volume}}{\text{Amount of sample (kg)}}$$

Results and discussion

Cypermethrin residues in soil and fruit samples

The concentrated extracts of eggplant fruit and the soil samples from the eggplant plot at different days after spraying (DAS) were subjected to analysis by GC-2010 with pre-set parameters. The results of the analysis of cypermethrin residues in eggplant fruit and soil samples are summarized in Table 1 and Table 2, respectively. The Food and Agriculture Organization of United Nations (FAO) has set up 0.5 ppm as the maximum residue limits (MRLs) for

insecticides in vegetables [8]. Above the MRLs of the insecticides are hazardous for human health.

From Table 1, it can be revealed that cypermethrin residues were detected (when applied at 1ml/L) in the eggplant fruit sample up to 5 DAS and the quantities were above the maximum residue limits (MRLs) up to 3 DAS. When cypermethrin was applied at the rate of 2 ml/L, residues were detected in the eggplant fruit samples up to 10 DAS. At 0 DAS, the residues in the fruit samples were 3.157 ppm, which degraded to 2.611 ppm at 1 DAS and 2.030 ppm at 3 DAS. At 5 DAS, it was 1.331 ppm and 0.768 ppm at 7 DAS which was still above the MRLs. After 10 DAS, the residues were 0.196 ppm, which was below MRLs level set by FAO.

Table 2 shows that cypermethrin residues were detected (when applied at 1ml/L) in the soil samples up to 5 DAS. At 0 DAS, the residues in the soil samples were 2.007 ppm, which degraded to 1.539 ppm at 1 DAS and 1.140 ppm at 3 DAS. After 5 DAS, it was 0.608 ppm and all the value was above the MRL. When cypermethrin was applied at the rate of 2 ml/L, residues were detected in the soil samples up to 7 DAS. At 0 DAS, the residues in the sample were 2.872 ppm, which degraded to 2.211 ppm at 1 DAS and 1.030 ppm at 3 DAS. After 5 DAS and 7 DAS, residues in the soil samples were 0.753 ppm and 0.351 ppm, respectively. But after 10 DAS no residues were detected.

Table 1 Quantity of cypermethrin residues in eggplant fruit samples.

Sample collection (Days)	Insecticide residues (ppm) 1 ml/L	Insecticide residues (ppm) 2 ml/L
0	2.575	3.157
1	1.961	2.611
3	0.762	2.030
5	0.031	1.331
7	0	0.768
10	0	0.196
12	0	0
15	0	0

Table 2 Quantity of cypermethrin residues in soil samples of the eggplant plot.

Sample collection (Days)	Insecticide residues (ppm) 1 ml/ L	Insecticide residues (ppm) 2 ml/L
0	2.007	2.872
1	1.539	2.211
3	1.140	1.030
5	0.608	0.753
7	0	0.351
10	0	0
12	0	0
15	0	0

Hossen [8] reported that cypermethrin residues were detected in tomato sample up to 5 DAS and the quantities were over MRLs up to 3 DAS, and this finding is quite comparable to the present study. Whereas cypermethrin residues in yard-long bean were found above the MRLs up to 5 DAS, reported by several researchers [9, 10].

To assure safety of the consumers, many of the developing countries have set Maximum Residue Limits (MRLs) based on Acceptable Daily Intake (ADI) and Potential Daily Intake (PDI) that should not be exceeded for a food item to be consumed safe for consumption [11]. It is assumed that the use of toxic pesticides on vegetables has raised the risk of intoxication to consumers along with diseases [12]. In Bangladesh, farmers have no idea about the pesticide residue level in the food as well as their effects and their levels are above MRLs. About 50-70% of the vegetables are contaminated with the residues of pesticides [13]. There is no insecticide available with the retention period less than three to five days [14].

Trend of residue degradation

Trend of degradation of cypermethrin residues in eggplant fruit and soil samples is shown in the Fig. 1 and 2. The Fig. 1 shows the cypermethrin residue degradation in eggplant fruits sprayed at the rate of 1 ml/L. The results revealed that 23.9% of cypermethrin residues were degraded at 1 DAS, which increased up to 70.4% at 3 DAS and 98.8% at 5 DAS. At 7 DAS, no residues were detected. When the cypermethrin was sprayed at 2 ml/L, residue degradation was up to 17.3% at 1 DAS, which increased gradually to 35.7% at 3 DAS, 57.8% at 5 DAS and 75.7% at 7 DAS. At 10 DAS, 93.9% residues were degraded and at 12 DAS no residues were detected.

Cypermethrin residue degradation in soil of the eggplant plot sprayed at 1ml/L can be revealed in Fig. 2. The results showed that 23.8% cypermethrin residues were degraded at 1 DAS, which gradually increased to 43.2% at 3 DAS and 69.7% at 5 DAS. At 7 DAS, no residue was found. When cypermethrin was sprayed at the rate of 2 ml/L, residues were degraded up to 23% at 1 DAS, which increased up to 64.1% at 3 DAS, 73.8% at 5 DAS and 87.8% at 7 DAS. The cypermethrin residues were not detected after 10 days.

Cypermethrin is relatively non-persistent in soil, and in sandy soil, it persists for 2-4 weeks. Increased cypermethrin persistence was observed in soil with high organic matter, high clay content, reduced microbial activity and anaerobic conditions [15]. The indiscriminate use of pesticides in rural and urban area of Bangladesh has contaminated agricultural producers, which affected soil health, surface water,

aquifers, wildlife, food and feeds all over the country [16-17].

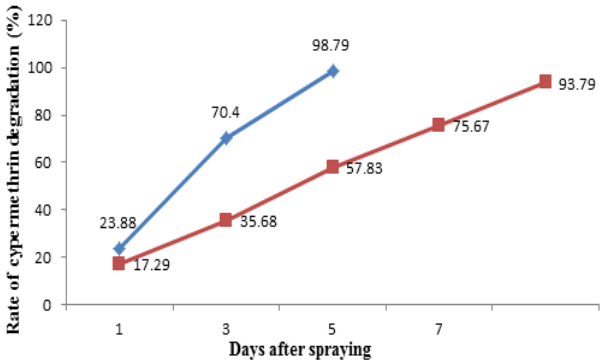

Fig. 1 Trend of cypermethrin residues degradation in eggplant fruit sprayed at 1 ml/L and 2 ml/L at different days after spraying.

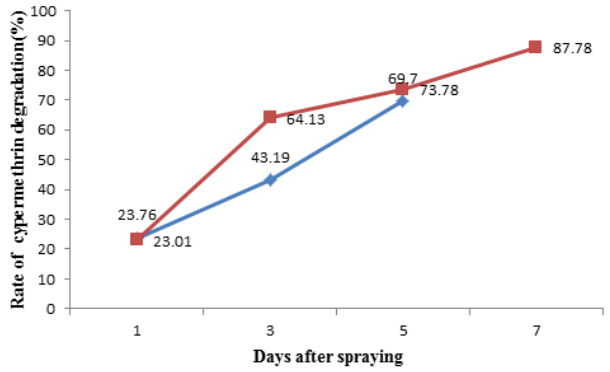

Fig. 2 Trend of cypermethrin residues degradation in the soil of eggplant plot sprayed at 1 ml/L and 2 ml/L at different days after spraying.

Conclusions

In this study, the cypermethrin residues were detected around 7 to 10 days after spraying and those were above the maximum residue limit level up to 5 days after spraying when the concentration of cypermethrin was 2 ml/L. Whereas, when the concentration of cypermethrin was 1 ml/L, the residues were detected up to 5 days after spraying and those were above the maximum residue limit level up to 3 days after spraying. As a result, it might be a suggestion to the farmers to use insecticide at a minimum rate to keep the environment safe and to the end users to consume eggplant fruit at least three and seven days after spraying of cypermethrin if it was treated with 1ml/L and 2ml/L, respectively.

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