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Effect of Processing on Dislodging of Cypermethrin Residues on Brinjal

Shweta Walia · Pinky Boora · Beena Kumari

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Abstract Brinjal fruits were sprayed with recommended dose (0.001%) of cypermethrin. Effects of processing viz. washing, cooking in water, cooking in oil, microwave cooking, and grilling was studied to dislodge cypermethrin residues on brinjal. Cypermethrin residues remaining in the control and processed samples weare analyzed by gas chromatography equipped with ECD. Dislodging of cypermethrin residues was observed more in grilling (50.12%), followed by cooking in oil (45.2%), cooking in water (41.4%), and microwave cooking (40.89%) after 1st day of the treatment. Reduction of residues after washing treatment was minimal.

Keywords Brinjal · Cypermethrin · Washing · Cooking · Microwave · Grilling · Pesticide residues · Gas chromatography

Vegetables, an important component of food is highly beneficial for the maintenance of human health and/or prevention of diseases (Hanif et al. 2006). Among vegetables, brinjal (*Solanum melongena*) is a common man's vegetable grown in almost all over India. After potato it ranks as the second highest consumed vegetable in India along with tomato (Mammoun et al. 2004).

Being most affordable, it is consumed as a main meal, soup, salad, vegetable, and curry preparations. Annually, India consumes about 8–9 million metric tons of brinjal, produced on 5.5 lakh hectares of land in different parts of the country. Rich in nutrition, brinjal has low calorific value, high water content, and provides adequate calcium, phosphorus, potassium, fibre, folic acid, sodium and vitamins B and C.

Besides several fungal diseases, brinjal is prone to attack by many insect pests, the most important being fruit and shoot borer, beetles, jassids, red spider mites, mealy bugs and aphids. Pesticide application is, therefore required to save the crop from pest infestation. Farmers extensively apply synthetic pyrethroids and organophosphorus insecticides to combat insect pests. On an average 4.6 kg of active ingredient of insecticide per hectare per season is applied on brinjal. It is the highest quantity applied to any vegetable after chilli which consumes about 5.13 kg of the insecticides. Among the various insecticides, cypermethrin, a low dosage insecticide, is used extensively for the control of various insect pests on vegetables in India. It degrades quickly in soil, get rapidly metabolized, and excreted by animals (Gill et al. 2001). Since farmers indiscriminately apply a cocktail of insecticides on vegetable crops, the increasing amount of pesticide residues in vegetables has been a major concern to the consumers. To ensure safe consumption, there is thus a need to develop methodologies to dislodge pesticide residues from brinjal. The present paper describes with the effect of washing and cooking processes (cooking in oil, cooking in water, microwave cooking and grilling) on dislodging of cypermethrin residues from brinjal.

Materials and Methods

The technical grade cypermethrin [α-cyano-3-phenoxybenzyl-*cis, trans*-3 (2,2-dichlovinyl)-2,2-dimethyl cyclopropane

S. Walia (✉) · P. Boora
Department of Foods and Nutrition, CCS Haryana Agricultural University, Hisar 125004, India
e-mail: shwetawalia28@gmail.com

B. Kumari
Department of Entomology, CCS Haryana Agricultural University, Hisar 125004, India

carboxylate] (93.0%), and its formulation (10% EC) was obtained from M/s Rallis India Limited, Bangalore. The stock solution (0.01%) of technical cypermethrin was prepared by dissolving 10.7 mg of the reference sample with 100 mL of the solvent. Similarly the stock solution (0.01%) of cypermethrin formulation was prepared by dissolving 10 mL of the formulation with 100 mL of water. Solution of the standard reference and the formulation (0.001%) was prepared by diluting 1 mL of the respective stock solution with 10 mL of the solvent.

Five kg marketable sized brinjal were procured from the control fields of the University farm where no pesticide has been sprayed in the last two seasons. Cypermethrin was sprayed on the fruits with 0.001% active ingredient up to drenching level. The treatments were in three replicates. The samples were kept at room temperature for 1 h and the fortified vegetable samples were processed and studied for the pesticide residue content after 1st, 2nd and 3rd day of treatment. The cypermethrin residue was determined by gas liquid chromatography (Shimadzu Model-2010) equipped with EC (^{63}Ni) detector, split injection system, and fused silica capillary column (SPB-5), 30 m × 0.32 mm 1D 0.25 μm film thickness of poly (5% diphenyl/95% dimethyl) silioxane as stationary phase, GLC operating conditions were: carrier gas flow, 60 ml min^{-1}, injector temperature, 230°C, detector temperature 290°C, oven temperature programme was 180°C (0 min) increasing @ 20°C min-1 up to 250°C (0 min), further increasing @ 5°C min^{-1} up to 280°C (10 min; Gill et al. 2001).

For determining the extent of dislodging and concentration of cypermethrin in brinjal fruits, the samples (20 g) were processed on 1st, 2nd and 3rd day after treatment. On each time interval, about half kg brinjal fruits were chopped into small pieces. After thorough mixing and quartering, 20 g representative sample was processed and extracted with 80 mL acetone after shaking for 1 h on a mechanical shaker. The content was filtered through the bed (2–3 cm) of anhydrous sodium sulphate and subjected to liquid partitioning thrice with hexane after diluting with 200 ml 10% aqueous sodium chloride by shaking vigorously for 1 min. Organic phase was collected and once again passed through anhydrous sodium sulphate. The organic phase was concentrated to 5 mL on a rotary vacuum evaporator at 40–45°C and cleaned by passing through a glass column compactly packed with aluminum oxide in between two layers of anhydrous sodium sulphate. After pre-wetting the column with 50 mL hexane, the residue was eluted with 100 mL hexane: acetone (9:1, v/v) mixture. The elute was concentrated on rotary vacuum evaporator to 5 mL (Mukherjee and Gopal 1992).

Prior to analysis of extracts for estimation of residues, recovery experiments were conducted. Brinjal fruits were spiked with 0.50 and 1.0 μg g^{-1} level of cypermethrin contents. 100 g of chopped brinjal was treated with 10 mL of 0.001% solutions of cypermethrin (tech.). All treatments were done in triplicate. Percentage recovery was calculated based on the difference between experimental and calculated values. Based on the results of recovery experiments, the optimum condition for extraction was adopted for the analysis of cypermethrin residues in controlled and treated samples. The concentration of each compound in the extracted vegetable was calculated by comparing the peak area with standard. At spiking levels of 0.50 and 1.0 μg g^{-1}, the respective recoveries recorded were 85.63 ± 3.01 and 82.11 ± 2.45% from brinjal. The limits of detection (LOD) of the GLC method were 0.02 μg g^{-1}.

To study the effect of washing, the whole fruits were washed under running tap water for 30 s. For cooking in boiling water, representative sample (20 g) was taken in conical flask with 20 ml water and cooked at 80–85°C for 10 min. While cooking brinjal fruits in oil, representative samples were cooked in 2 mL vegetable oil for 3 min. In two different experiments, representative samples (28 g) were micro-waved, and grilled for 3 and 5 min, respectively. The processed samples were extracted, cleaned and analyzed by GC in a similar way employed for fresh samples.

The experiments were set up in a completely randomized design. All data were analyzed by analysis of variance (ANOVA). All the experiments were repeated thrice as means ± standard error of mean.

Results and Discussion

The study revealed that under laboratory conditions (Table 1), the initial cypermethrin deposits of 1.570 on first day declined to 0.956 and 0.750 μg g^{-1} in 2 and 3 days, showing 39.10 and 52.22% dissipation, respectively. The similar trend in the decline of initial deposits of synthetic pyrethroids on brinjal has been reported earlier (Metwally et al. 1997; Gill et al. 2001). The degradation process was quite low, indicated the prolonged persistent nature of the insecticide.

The data on dislodging of cypermethrin residues following culinary and cooking processes is given in Table 1. Residues of cypermethrin following washing were reduced to the level of 1.170 from 1.570 μg g^{-1} on the first day, 0.727 μg g^{-1} on the second, and 0.593 μg g^{-1}, on the third day. Percent reduction in residues due to washing was 25.47, 23.95 and 20.93 on the corresponding days. In earlier reports, washing resulted in 42.2 and 35.7% reduction of β-cyfluthrin residues on okra (Dikshit et al. 2002), and 39.0 and 30.0% reduction of lamda cyhalothrin residues on tomato on 0 day and 3rd day, respectively (Jayakrishnan et al. 2005).

Table 1 Effect of processing on dislodging of cypermethrin residues in/on brinjal

Process	Residues (μg g^{-1})		
	1st day	2nd day	3rd day
Unprocessed (control)	1.570 ± 0.105	0.956 ± 0.145 (39.10)	0.750 ± 0.019 (52.22)
Microwave cooking	0.928 ± 0.025 (40.89)	0.601 ± 0.095 (37.13)	0.488 ± 0.076 (34.93)
Cooking in water (boiling)	0.920 ± 0.505 (41.40)	0.579 ± 0.042 (34.43)	0.477 ± 0.043 (36.40)
Cooking in oil (frying)	0.860 ± 0.031 (45.22)	0.545 ± 0.032 (42.99)	0.446 ± 0.016 (40.53)
Grilling	0.783 ± 0.035 (50.12)	0.495 ± 0.038 (48.22)	0.405 ± 0.047 (46.00)
Washing	1.170 ± 0.013 (25.47)	0.727 ± 0.005 (23.95)	0.593 ± 0.030 (20.93)
CD ($p \leq 0.05$)	0.39	NS	NS

Values are mean ± SE of three replicates in μg g^{-1}

Figures in parentheses indicate per cent removal/dislodging

NS Non-significant, *CD* Critical difference

Following cooking of brinjal in boiled water, residues of cypermethrin were reduced to 0.920 from 1.570 μg g^{-1} on the first day which further declined to 0.579 and 0.477 μg g^{-1} showing 41.40, 34.43 and 36.40% reduction on the corresponding days, respectively. Similar results were obtained earlier by Dikshit et al. (2002) who reported that there was 41.3% β-cypermethrin residues reduction on day 0 and 34.6% residue reduction on 3rd day on cooking of sample fruits. Cooking of brinjal fruits in oil resulted in reduction of residues to 0.860 from 1.570 μg g^{-1} on the first day. The residues were further reduced to 0.545 and 0.466 μg g^{-1} on the second and third day, respectively. The rate of dislodging of residues due to cooking depends upon factors like temperature, duration of the process, the amount of water, food additives and the type of system (open/closed).

Since many brinjal dishes are based on high temperature cooking, experiments were conducted to study the effect of microwave cooking and grilling on the cypermethrin residues on brinjal. Following microwave cooking, residues of cypermethrin were reduced to 0.928 from 1.570 μg g^{-1} on the first day. On the 2nd and 3rd day, the residue was further reduced to 0.601 and 0.488 μg g^{-1}, respectively showing 40.89, 37.13 and 34.93% reduction in the residues. Studies were also conducted to see the effect of grilling on the dislodging of cypermethrin residues on brinjal. Results revealed that as compared to initial concentration of 1.57 μg g^{-1}, the residue levels after grilling operation reached the lower levels of 0.783, 0.495 and 0.405 μg g^{-1} indicating 50.12, 48.22 and 46.00% reduction on the 1st, 2nd and 3rd day, respectively.

Statistical analysis (CD, $p \leq 0.05$) proved that decontamination of cypermethrin residues by various cooking processes was significant as compared to control samples. Grilling process appeared to be the most effective. However, with passage of time, the effectiveness of such decontamination processes is reduced as the lipophilic insecticide goes inside the waxy coat of brinjal fruit skin. Accordingly the dislodgable residues are less on 2nd and 3rd day as compared to day one sample. Similar results have been reported earlier on another synthetic pyrethroid cyfluthrin (Sinha and Gopal 2002). While the loss in unprocessed (control) field sample is due to abiotic factors, the processing losses on day-1 for all the treatment are statistically significant.

Farmers resort to repeated application of insecticides such as cypermethrin to combat insect infestation on brinjal crop. Excessive residues remaining on brinjal therefore need to be decontaminated before consumption of brinjal recipes. Effects of processing viz. washing, cooking in oil, microwave cooking and grilling has been studied to dislodge cypermethrin residues on brinjal. The overall results indicated that cypermethrin residues can be more effectively dislodged by grilling process followed by cooking in oil, cooking in water and micro-wave cooking. Reduction of residues was however, minimum after washing. Cooking processes thus lead to significant destruction of cypermethrin residues.

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