Bull Environ Contam Toxicol (2011) 87:693–698
DOI 10.1007/s00128-011-0395-8

Persistence of Cypermethrin and Decamethrin Residues in/on Brinjal Fruits

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Received: 20 April 2011 / Accepted: 25 August 2011 / Published online: 25 September 2011

Abstract Residues of cypermethrin and decamethrin were estimated in brinjal fruits by gas liquid chromatography following single application of Cymbush 25 EC @ 43.75 and 87.50 g a.i./ha and of Decis 2.8 EC @ 11.20 and 22.40 g a.i./ha at fruiting stage. The average initial deposits of cypermethrin 0.600 and 1.095 mg kg^{-1} and of decamethrin 0.430 and 0.900 mg kg^{-1} were observed for single and double dose, respectively. Residues reached below maximum residue limit (MRL) value of 0.2 and 0.05 mg kg^{-1} on third and seventh day for cypermethrin and decamethrin, respectively. The half-life values ($t_{1/2}$) were worked out to be 1.16, 1.18 days for cypermethrin and 1.33, 1.42 days for decamethrin at single and double dose, respectively following first order kinetics. Washing and washing followed by boiling/cooking processes were found to be effective in reducing the residues of both the insecticides in brinjal fruits. Maximum reduction (31–42%) and (26–37%) was observed by washing followed by boiling/cooking for cypermethrin and decamethrin, respectively.

Keywords Cypermethrin · Decamethrin · Brinjal · Residues · Half-life period · Processing

Vegetables are the fresh and edible portion of the herbaceous plants and essential components of human diet. These are highly beneficial for the maintenance of human health and prevention of diseases (Hanif et al. 2004). But for better yield and quality, insecticides are repeatedly applied during the entire period of growth and sometimes even at the fruiting stage. About 10–12% of the total pesticides are used on fruit and vegetable crops. Their persistent use leads to build up of toxic residues on crop produce, which may exert adverse effects on human health in addition to disturbing ecosystem (Kumari et al. 2003). This problem is more prevalent in vegetables as they absorb insecticides and often create health hazards to human beings while consuming fresh or without much processing. Among vegetables, brinjal or egg plant (*Solanum melongena* L., aubergine) is one of the important vegetable grown and consumed in India and other tropical countries. After potato, it ranks as the second highest consumed vegetable in India along with tomato (Mammoun et al. 2004). In 2008–2009, total area under brinjal cultivation was 0.6 million ha with production of 10.37 million tonnes. This crop is vulnerable to attack from several insect pests like jassids, white flies, caterpillars, etc., *Leucinodes orbonalis* G. (shoot and fruit borer) is considered to be the key pest. Management of this insect pest of brinjal plant is difficult since it harbors inside the shoot and fruit portions of eggplant (Nair 1986; Regupathy et al. 1989; Sardana et al. 2004). Several insecticides belonging to organochlorine, organophosphorus, cyclodienes, and synthetic pyrethroids have been evaluated to manage this pest (Mukherjee and Gopal 1992). Now a days, mostly pyrethroids are used for insect-pest control because they degrade quickly in soil, get rapidly metabolized, virtually have zero mobility in soil and excreted by animals. Among the various insecticides, cypermethrin and decamethrin are low dose insecticides used extensively for the control of various insect pests on vegetables in India. Since farmers indiscriminately apply a cocktail of insecticides on vegetable crops, thus the increasing amount of pesticide residues in vegetables has been a major concern

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Table 1 Residues (mg kg^{-1}) of cypermethrin in brinjal

Days after treatment	Single dose (43.75 g a.i./ha)		Double dose (87.50 g a.i./ha)		Mean
	Average residues[a] (mg kg^{-1}) ± SD	Dissipation (%)	Average residues (mg kg^{-1}) ± SD	Dissipation (%)	
0	0.600 ± 0.015	–	1.095 ± 0.081	–	0.848
3	0.100 ± 0.002	83.33	0.189 ± 0.0008	82.73	0.145
7	0.043 ± 0.004	92.83	0.084 ± 0.006	92.32	0.064
10	0.030 ± 0.003	95.00	0.065 ± 0.005	94.06	0.048
Mean	0.193		0.358		
	Correlation coefficient $r = -0.9456$; $t_{1/2} = 1.16$ day		Correlation coefficient $r = -0.9357$; $t_{1/2} = 1.18$ day		

CD ($p \geq 0.05$) for days = 0.009, for dose = 0.006, for days × for dose = 0.012

[a] Average of three replicates; MRL: 0.2 mg kg^{-1}

to the consumers. To ensure safe consumption of food commodities, there is a need to develop methodologies to dislodge pesticide residues from them. The present study was undertaken to study the persistence behaviour of these two pyrethroids following spray application and effect of kitchen processes like washing and washing followed by boiling/cooking on reduction of residues of cypermethrin and decamethrin from brinjals fruit.

Materials and Methods

Brinjal (Variety BR 112) crop was raised and transplanted in a plot size of 2.4 m × 2.1 m according to the recommended agronomical practices in the Research Field of Entomology, CCS Haryana Agricultural University, Hisar during *Kharif season* (summer season, from April to November) 2009–2010. Cypermethrin (Cymbush 25 EC) at the rate of 43.75 (T_1) and 87.50 (T_2) and decamethrin (Decis 2.8 EC) at the rate of 11.20 (T_1) and 22.40 (T_2) g active ingredient (a.i.) ha^{-1} were applied in triplicate along with control in randomised block design (RBD) at fruiting stage of brinjal crop. In control plots, only water was sprayed.

Brinjal fruit (1 kg) samples were collected randomly on 0 day (1 h after spray), 3, 7, and 10 days after treatment in three replicates from both the trials. Samples collected from field were analyzed at three stages i.e. raw, after washing and washing followed by boiling/cooking to determine residues of cypermethrin and decamethrin. The composite brinjal samples were divided into three potions, one portion was processed as such second after washing and third one after washing followed by boiling/cooking. Washing was performed by placing brinjal fruits in a container and rinsed under tap water for 30 s, with gentle rotation by hand as described by Walter et al. (2000) and blotted dry with a paper towel and divided into two parts. One part was used for washing and second for boiling. For boiling/cooking, in 25 g washed sample of brinjal 10 mL water was added and boiled till softness of fruit pieces. Effect of processing was studied in 0 and 3 days samples only.

All the solvents (hexane, acetone, n-hexane) were procured from Merck and glass distilled before use. Activated charcoal and neutral alumina were of Analytical Grade. Technical standard of cypermethrin and decamethrin were procured form Sigma-Aldrich.

Extraction and clean-up was performed as per method of Dikshit et al. (2001). Representative 25 g of the finely chopped sample was extracted with 100 mL acetone by shaking on mechanical shaker for 1.5 h. Filtered the extract and partitioned thrice with hexane in separatory funnel

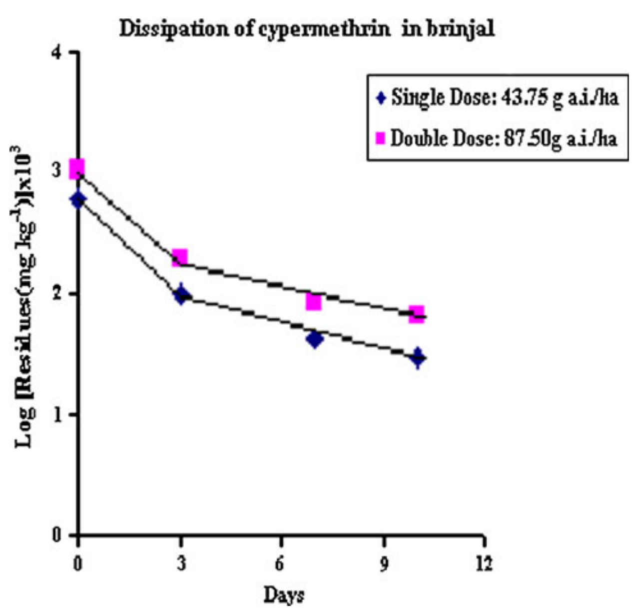

Fig. 1 Linear plot for first order kinetics of cypermethrin dissipation in brinjal

Table 2 Residues (mg kg^{-1}) of decamethrin in brinjal

Days after treatment	Single dose (11.20 g a.i. ha^{-1})		Double dose (22.40 g a.i.ha^{-1})		Mean
	Average residues[a] (mg kg^{-1}) ± SD	Dissipation (%)	Average residues (mgkg^{-1}) ± SD	Dissipation (%)	
0	0.430 ± 0.008	–	0.900 ± 0.005	–	0.665
3	0.090 ± 0.007	79.06	0.210 ± 0.008	76.66	0.150
7	0.005 ± 0.001	98.83	0.022 ± 0.002	97.55	0.014
10	0.003 ± 0.001	99.30	0.010 ± 0.004	98.88	0.007
Mean	0.132		0.286		
	Correlation coefficient r = −0.9810; $t_{1/2}$ = 1.33 day		Correlation coefficient r = −0.9921; $t_{1/2}$ = 1.42 day		

CD ($p \geq 0.05$) for days = 0.018, for dose = 0.013, for days × for dose = 0.025

[a] Average of three replicates; MRL: 0.05 mg kg^{-1}

after diluting with 10% solution of sodium chloride. The organic layers were pooled and concentrated on rotary vacuum evaporator to reduce the volume approximately to 10 mL. Glass column (60 cm × 22 mm i.d.) was packed compactly with activated charcoal and neutral alumina (5:1 w/w). Prewetted the column with 40 mL of hexane, loaded the concentrated extract in the column and eluted with 100 mL solution of hexane: acetone (1:1 v/v) at flow rate of 4 mL/min. Concentrated the eluate on vacuum evaporator followed by gas manifold evaporator. Final volume was made to 2 mL in n-hexane for GC analysis.

A Shimadzu Gas Chromatograph GC-2010 equipped with electron capture detector (ECD) ^{63}Ni and a capillary column HP-1 (30 m × 0.32 mm i.d. × 0.25 µm film thickness of 95% dimethyl and 5% diphenyl polysiloxane) was used for residue analysis of cypermethrin and decamethrin. The operating parameters of the instrument were: Oven temperatures (°C) 150 (5 min) → 8°C min^{-1}→190 (2 min) → 15°C min^{-1} → 280 (10 min), injection port 280°C and detector 300°C. Flow rate of nitrogen (carrier gas) was 60 mL min^{-1}, 2 mL min^{-1} through column and split ratio 1:10. Under these operating conditions, the retention times of cypermethrin were observed as 20.834, 20.955, 21.082 min and for decamethrin 23.597 min.

Results and Discussion

The percent recoveries at the fortification levels of 0.25 and 0.50 mg kg^{-1} of cypermethrin and decamethrin were in the range of 87.11–89.63 and 95.10–91.57, respectively. The residue data of cypermethrin on brinjal are presented in Table 1. The experimental data reveled that the initial deposit for cypermethrin at single and double dose were 0.600 and 1.095 mg kg^{-1}, respectively. The residues dissipated with time by 83.33% and 82.73% in 3 days at respective doses. After 3 days, there was slow dissipation of cypermethrin residues till 10 days.

Although the dissipation rate was slightly slow but kept on increasing day by day and about 95% of residues dissipated after 10 days with half-life period of 1.16 and 1.18 days for single and double doses, respectively. The dissipation pattern followed the biphasic first order kinetics (Fig. 1). Residues of cypermethrin from both the doses reached below maximum residue limit (MRL) of 0.2 mg kg^{-1} in 3 days. Hence forth from consumer's health point of view, a safe waiting period of 3 days is suggested. Statistically analysed data using ANOVA showed that irrespective of the duration, at single dose, significantly less residues (0.193 mg kg^{-1}) were recorded than the double dose (0.358 mg kg^{-1}) (CD = 0.006; $p = 0.05$). With increase in duration, level of residues decreased significantly (CD = 0.009; $p = 0.05$). Interaction between duration and doses were also significant (CD = 0.012; $p = 0.05$). This suggested that single dose has

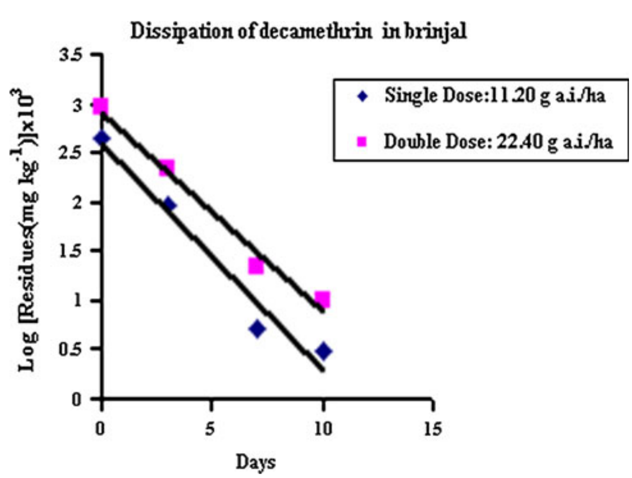

Fig. 2 Linear plot for first order kinetics of decamethrin dissipation in brinjal

Table 3 Effect of processing on residues (mg kg^{-1}) of cypermethrin and decamethrin in brinjal

Days after treatment	Single dose (43.75 g a.i.ha^{-1})			Double dose (87.50 g a.i.ha^{-1})		
	Initial deposit (mg kg^{-1})[a] ± SD	W	W + C	Initial deposit (mg kg^{-1})[a] ± SD	W	W + C
Cypermethrin						
0	0.600 ± 0.015	0.390 ± 0.004 (35.00)	0.350 ± 0.010 (41.66)	1.095 ± 0.081	0.729 ± 0.013 (33.42)	0.690 ± 0.007 (36.98)
3	0.100 ± 0.002	0.072 ± 0.001 (28.00)	0.064 ± 0.007 (36.00)	0.189 ± 0.008	0.142 ± 0.021 (24.86)	0.131 ± 0.024 (30.68)
Days after treatment	Single dose (11.20 g a.i. ha^{-1})			Double dose (22.40 g a.i. ha^{-1})		
	Initial deposit (mg kg^{-1})[a] ± SD	W	W + C	Initial deposit (mg kg^{-1})[a] ± SD	W	W + C
Decamethrin						
0	0.430 ± 0.008	0.310 ± 0.001 (27.90)	0.270 ± 0.025 (37.20)	0.900 ± 0.005	0.675 ± 0.007 (25.00)	0.607 ± 0.006 (32.55)
3	0.090 ± 0.007	0.070 ± 0.002 (22.22)	0.062 ± 0.002 (31.11)	0.210 ± 0.008	0.167 ± 0.001 (20.47)	0.156 ± 0.005 (25.71)

Figures in parenthesis are the percent reduction of residues

[a] Average of three replicates; *W* washing; *W + C* washing followed by boiling/cooking

significantly less residues as compared to double dose and with increase in duration, level of residues decreased significantly in both the doses by 10 days from brinjal fruits. Singh and Kalra (1992) reported that initial deposit of cypermethrin (@ 100 g a.i. ha^{-1}) was 0.73 mg kg^{-1} in brinjal which declined to 0.61 mg kg^{-1}, 1 day after treatment and 0.08 mg kg^{-1} after 10 days of spraying. They also reported a dissipation of 94% which is in conformity with the present findings.

Residue data pertaining to decamethrin have been presented in Table 2. As clear from the data, initial deposit of 0.430 mg kg^{-1} at the rate of 11.20 g a.i. ha^{-1} dissipated to the level of 0.090 mg kg^{-1} after 3 days showing 79.06% dissipation whereas at the rate of 22.40 g a.i. ha^{-1}, almost same extent of dissipation (76.66%) was observed when the initial deposit of 0.900 mg kg^{-1} dissipated to the level of 0.210 mg kg^{-1}.

Residues reached below MRL value of 0.05 mg kg^{-1} after 7 days in both the doses. The insecticide dissipated very fast just after its application in both the doses and from third day onwards there was a gradual degradation/dissipation of decamethrin residues till 10 days. With in this period, 99.30% and 98.88% dissipation was recorded in respective doses following a first order kinetics (Fig. 2) with half life period of 1.33 and 1.42 days. Statistically analysed data using ANOVA showed that irrespective of the duration at single dose, significantly less residues (0.132 mg kg^{-1}) were recorded than the double dose (0.286 mg kg^{-1}) (CD = 0.013; $p = 0.05$). With increase in duration, level of residues also decreased significantly (CD = 0.018; $p = 0.05$). This suggested that single dose has significantly less residues as compared to double dose and with increase in duration, level of residues significantly decreased in both the doses. Mondal et al. (1987) reported that the initial deposits of decamethrin when applied @ 0.02% and 0.04% varied from 0.046 to 0.090 ppm and declined to 0.002–0.005 ppm on tenth day in brinjal fruits which is in conformity with the present results. Raha et al. (1993) reported first order kinetics and half life values of decamethrin applied @ 0.0015% and 0.0030% on brinjal fruits were 1.20–4.30 days which is also in confirmation with present results.

Effect of Processing

Brinjal fruits were subjected to processing like washing, washing followed by boiling/cooking in order to investigate the reduction of residues of cypermethrin and decamethrin from 0 to 3 days samples. The data pertaining to this experiment is presented in Table 3.

It has been observed that washing of 0 day brinjal fruits with water reduced 33.42–35.00% and 25.00–27.90% residues of cypermethrin and decamethrin in both the treatments, respectively. The corresponding reduction in the residues due to washing followed by boiling/cooking was 36.98–41.66% and 32.55–37.20%. On third day, effect of washing as well as washing followed by boiling was comparatively less effective in reducing the residues of both insecticides. This may be due to less availability of residues on surface as with passage of time, surface residues become less available due to penetration in the fruit.

From the results it has been concluded that both the processes used in this study were more effective in reducing the residues on 0 day because of availability of residues on surface which could be dislodged easily. Raha et al. (1993) reported that washing of brinjal fruits under tap water removed decamethrin residues to an extent of 29–50% while washing followed by cooking reduced deposits by 50–74% which are quite similar with the present findings.

Reduction of cypermethrin residues on brinjal to the level of 26% by washing and 37% by washing followed by boiling was reported by Kumari (2008). Walia et al. (2010) reported that washing of brinjal fruits under tap water removed cypermethrin residues to an extent of 39.10% on 2nd day which are in conformation with the present results. The similar trend in the decline of initial deposits of synthetic pyrethroids on brinjal has been reported earlier by Metwally et al. (1997) and Gill et al. (2001). The overall results indicated that cypermethrin and decamethrin residues can be more effectively dislodged by washing followed by boiling/cooking.

Acknowledgments The authors are grateful to the Head, Department of Entomology, CCS Haryana Agricultural University, Hisar, for providing necessary research facilities.

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