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Effect of processing on acetamiprid residues in eggplant fruits, *Solanum melongena* L.

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An experiment was conducted to find out the persistence and the effect of processing on acetamiprid, namely 20% soluble powder (SP) in eggplant fruits via washing, cooking in water, oil, and grilling. Acetamiprid half-life values in eggplant fruits, leaves and soil were found to be 1.96, 2.31 and 10.47 days, respectively. A higher removal rate was found in boiling and grilling (56 and 99% than in frying (46.24%) and washing (24.73%) after the first day of the treatment. The results show that acetamiprid at the recommended dosage can be considered quite safe from the point of view of health hazards to consumers and a waiting period of one day is suggested to reduce the risk before consumption of eggplant fruits and processing products.

Key words: Eggplant, acetamiprid, washing, cooking, grilling, pesticide residues.

INTRODUCTION

Eggplant (*Solanum melongena* L.) is one of the important vegetables grown and consumed in Egypt and other tropical countries. Eggplant fruit is ranked amongst the top ten vegetables in terms of oxygen radical absorbance capacity due to the fruit phenolic component (Stommel and Whitaker, 2003). Eggplants which are low in calories, have high water content, and provides relatively high levels of calcium, phosphorus, potassium, fiber, folic acid, sodium and vitamins B and C (Kashyap et al., 2003). Eggplant fruits are vulnerable to attacks from several insect pests like shoot borer, beetles, jassids, red spider mites, mealy bugs and aphids. Pesticide application is therefore required to save the crop from pest infestation (Sardana et al., 2004). Farmers however, apply different pesticides for controlling them.

In the Nineties, acetamiprid {(E)-N1-[(6-chloro-3-pyridyl) methyl]-N2-cyano-N1-methylacetamidine} was registered in Japan, New Zealand, China and other European countries (Gupta et al., 2008). Due to its special acting mechanism, high efficacy, and relatively low toxicity, and no evidence of carcinogenicity, neurotoxicity, mutagenicity or endocrine disruption, acetamiprid is being more competitive and has been considered as a favorable alternative to the organophosphate insecticides, which have caused severe environmental pollution and banned in many countries (U.S. Environmental Protection Agency, 2002). It was reported that the half-life value of acetamiprid was 1.02-1.59 days in the mustard plant (Pramanik et al., 2006); 1.82 to 2.33 days in tea (Sharma et al., 2008); 2.24 days in chili (Sanyal et al., 2008) and 1.9 to 2.5 days from Zucchini and Zucchini leaves (Park et al., 2011). However, no information is currently available concerning the residual status and pattern of acetamiprid degradation and removal in fresh or processed eggplants. The main objective of this study was firstly, to determine the persistence, pattern of decline and residue levels of acetamiprid in fresh and processed (grilled, boiled, washed, fried) eggplant fruits and secondly, to demonstrate if acetamiprid poses any residual toxicity

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problem in eggplant fruits.

MATERIALS AND METHODS

Sampling

A field experiment was conducted at the Aboutouala, Mania El-kamh province, Sharkia governorate, Egypt, on 15 June, 2011. Eggplant (*S. melongena* L.) plants were cultivated in plots consisting of 10 rows separated by a three row belt. The experiment was conducted in a randomized block design using three replicates for each treatment. Common agricultural and fertilization practices were used. One formulation of Acetamiprid namely 20% SP (soluble powder) was examined for their persistence behavior on the eggplant fruit and their residues in leaves and soil.

An aqueous solution of the formulations were applied at the rates of 20 g ai / per feddan (5 g ai /100 L water), (1 feddan = 4, 200 m^2) which corresponds with the recommended dose. The application was done using a knapsack sprayer. The control plots were left unsprayed. Samples of eggplant fruits (1 kg), from both treated and control plots were collected randomly from different heights of the plants at different time interval (0, 1, 3, 5 and 7 days) after the application. Soil samples of 1 kg were collected from the surface of the plots to a depth of 10 cm after an hour and 1, 3, 5, and 7 days after treatment. Also, samples of eggplant leaves (50 g) were taken at the same periods from corresponding plots.

Washing and cooking processes

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 to 85°C for 10 min. While cooking eggplant fruits in oil, representative samples were cooked in 20 ml vegetable oil for 3 min. In a different experiment, representative sample (28 g) was grilled for 5 min. The processed samples were extracted, cleaned and analyzed by High-Performance Liquid Chromatography (HPLC) equipped with UV detector in a similar way employed for fresh samples.

Extraction and cleanup

Acetamiprid was extracted and cleaned up from eggplant fruits (20 g), leaves (20 g) and soil (50 g) according to the method described by Sanyal et al. (2008). The residues of acetamiprid were estimated by high pressure liquid chromatography with UV detector. The detector was set at 246 nm wavelength. A C18 column was used, and the mobile phase was a mixture of Acetonitril/Water (40:60 v/v) at 1.0 ml min^{-1} flow rate. The retention time was 2.83 min. To validate the method, prior to analysis of extracts for estimation of residues, recovery experiments were carried out at 1, 5 and 10 Limit of Quantification, LOQ (0.062, 0.31 and 0.62 µg g^{-1}). Each 0.124, 0.62 and 1.24 ml of 10 µg ml^{-1} standard solution of acetamiprid were added to the respective homogenized control sub sample (20 g) of eggplant fruits, leaf and soil.

All determinations were done in triplicates. 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 acetamiprid residues in controlled and treated samples. The concentration of each compound in the extracted vegetable was calculated by comparing the peak area with standard deviation. In this study, all statistical analyses were performed with CoStat 6.311 (Cohort Software). The rate of degradation (K) and Half-life (t 1/2) were obtained from the following equation of Gomaa and Belal, (1975):

$$\text{Rate of degradation (K)} = 2.303 \times \text{slope} \quad (1)$$

$$\text{Half-life (t 1/2)} = 0.693/K \quad (2)$$

RESULTS AND DISCUSSION

Method validation

The calibration curve was obtained by plotting peak areas in 'y' axis against concentrations of the pesticide in 'x' axis within the investigated range (0.75 - 6.25 µg ml^{-1}) of concentrations. Each solution was injected in triplicates. The linear range, intercept and slope of the curve were found to be 0.0.75 - 6.25 µg ml^{-1}, 15.54 and 28.16, respectively. The linearity was good with a high correlation coefficient of $r^2 = 0.998$. The Limit of Detection (LOD) and Quantification (LOQ) were evaluated using the following equation:

$$\text{LOD} = 3.3\ S0/b \text{ and } \text{LOQ} = 10S0/b \quad (3)$$

Where S_0 is the standard deviation of the calibration line and b is the slope (Thomsen et al., 2003). The Limit of Detection LOD and Quantification LOQ of acetamiprid in this study were found to be 0.411 µg ml^{-1} corresponding to 0.021 mg kg^{-1} and 1.25 µg ml^{-1} corresponding to 0.062 mg kg^{-1}, respectively. This method provides a detection limit which is lower than the maximum residue limit (MRL) established by the European Union (Europe Commission, 2010) for acetamiprid in eggplant fruit (0.1 mg kg^{-1}). The precision values for the method, expressed as relative standard deviation (RSD), was 0.765 (n=3) for acetamiprid. Insecticide recovery ranged from 91.00 to 92.45% in fruits, from 85.48 to 90.32% in leaves and from 90.32 to 91.93% in soil samples (Table 1).

Incecticide degradation under field conditions

The persistence of acetamiprid in eggplant fruit leaves and soil samples at different time points are shown in Table 2. The initial deposits (1 h after spraying) of acetamiprid in eggplant fruits, leaves and soil were 0.143, 0.170 and 0.862 µg g^{-1}, respectively. As expected no residue was detected in the untreated control samples. The residues of acetamiprid in eggplant fruits, leaves and soil samples declined progressively with time. About 66.43, 42.35 and 14.39% of the initial residue was dissipated 3 days after of application. After 5 days the residues levels were 77.62, 84.71 and 26.91% for all application doses. No residues were detected after one week. The dissipation of acetamiprid followed first order reaction kinetics. The calculated half-life values of acetamiprid in eggplant fruits, leaves and soil were 1.96,

Table 1. Average recovery percentage and relative standard deviation at different fortification levels.

Substrate	Fortification level (µg/g)	Recovery (%) (Mean ± SD) *
	0.062	91.00± 0.76
Eggplant fruits	0.31	91.89± 0.70
	0.62	92.45±1.50
Mean	-	91.78±0.99
	0.062	85.48±1.9
Leaves	0.31	87.09±2.0
	0.62	90.32±2.49
Mean	-	87.63±2.13
	0.062	90.32±2.05
Soil	0.31	90.64±3.52
	0.62	91.93±3.98
Mean	-	90.96±3.18

* Each value is mean ± SD of three replicate determinations.

Table 2. Residues of acetamiprid in eggplant.

Days	Mean (µg g^{-1}) ± SD (% dissipation)*		
	Eggplant fruits	Leaves	Soil
Initial**	0.143± 0.03 (0.00)	0.170± 0.03 (0.00)	0.862±0.05 (0.00)
1	0.093± 0.02 (39.96)	0.128± 0.02 (24.71)	0.776±0.04 (9.98)
3	0.048 ± 0.01 (66.43)	0.098± 0.02 (42.35)	0.738±0.04 (14.39)
5	0.032± 0.01 (77.62)	0.026± 0.01 (84.71)	0.630±0.03 (26.91)
7	N. D	N. D	0.518 ± 0.02 (39.91)
Half life $T_{1/2}$ (days) 1.96		2.31	10.47

*Average of three replications; Values in parenthesis are % dissipation in comparison to 0 day ** one hour post spraying.

2.31 and 10.47 days, respectively (Table 2). Pesticide dissipation depends on physical and chemical factors, including environmental conditions, mode of application, plant species and growth rate, dosage, interval between applications and time of harvest (Khay et al., 2008). It has been reported that the half-life value of acetamiprid was 1.02-1.59 days in the Mustard plant (Pramanik et al., 2006); 1.82 to 2.33 days in Tea (Sharma et al., 2008); 2.24 days in Chili (Sanyal et al., 2008) and 1.9 to 2.5 days in Zucchini fruits and leaves (Park et al., 2011).

In the present work, the persistence of acetamiprid was higher in soil than in fruit and leaves. The dissipation decreased in soil through 1 to 3 days but subsequently increased later on (Table 2). This may be associated with irrigation of eggplant after 4 days of treatment. The longer persistence, and hence slower dissipation, under air-dry conditions could be attributed to the low microbial activity in dry soil (Gupta and Gajbhiye, 2002). The data on dislodging of acetamiprid residues following washing in water, cooking in oil and water, and grilling processes are shown in Table 3. Residues of acetamiprid following washing were reduced to 0.07 after 1 day.

One fold reduction was found between days 1 and 3. Finally after 5 days, only 0.025 µg g^{-} were found. Residue reduction due to washing was 24.73, 22.92 and 21.87 after 1, 3 and 5 days respectively. Rinsing of various vegetables was found very effective to eliminated residues of some pesticides (Jayakrishnan et al., 2005). Further processing techniques could also contribute to the decrease in the concentration of pesticide residues in the vegetable and fruit samples (Shweta et al., 2010). Acetamiprid removal depends on the physical and chemical properties of the pesticide, method of application, as well as the nature of the cultivated plant. Residues may get dried on the surface, adsorbed to waxy material in the outer portion of the fruit or vegetable, or translocated into the inner tissues of the plant with reduction in the removal of the active principle (Ripley and Edgington, 1983).

After boiling, acetamiprid residues were reduced to 0.04 on the first day. The residue levels decreased to below detectable limit on the fifth day of treatment.

Table 3. Effect of processing on dislodging of acetamiprid residues in/on eggplant process residues (µg g^{-1}).

Parameter	1st day	3rd day	5th day
Unprocessed (control)	0.093±0.02[a] (0.00)	0.048 ± 0.01[a] (48.38)	0.032± 0.01[a] (65.59)
Washing	0.07 ± 0.02[b] (24.73)	0.037± 0.01[b] (22.92)	0.025±0.00[b] (21.87)
Cooking in water (boiling)	0.04 ± 0.01[d] (56.99)	0.029 ± 0.00[d] (39.58)	0.00[c]
Cooking in oil (frying)	0.05± 0.02[c] (46.24)	0.033± 0.01[c] (31.25)	0.023±0.01[b] (28.13)
Grilling	0.04± 0.01[d] (56.99)	0.030± 0.01[cd] (37.50)	0.00[c]
Significance	***	***	***

Values are mean ± SD of three replicates in µg g^{-1}. Figures in parentheses indicate percent removal/dislodging.*** High significance.

Following frying the residues of acetamiprid was reduced to 0.050 from 0.093 µg g^{-1} on the first day. On the 3rd and 5th day, the residue was further reduced to 0.033 and 0.023 µg g^{-1}, respectively showing 46.24, 31.25 and 28.13% reduction in the residues. 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) (Angioni et al., 2004). The processes that normally occur during cooking are volatilization, hydrolysis and thermal breakdown (Balinova et al., 2006). Since many eggplant dishes are based on high temperature cooking, experiments were conducted to study the effect of grilling on the acetamiprid residues on eggplant fruits. Studies were also conducted to see the effect of grilling on the dislodging of acetamiprid residues on eggplant fruits. Results revealed that as compared to initial concentration of 0.093 µg g^{-1}, the residue levels after grilling operation reached 0.040 and 0.030 µg g^{-1} indicating 56.99 and 37.50% reduction on day 1 and 3, respectively.

The residue levels decreased to below detectable limit on the 5th day of treatment. Boiling and grilling processes were the most effective. However, as time progresses, the effectiveness of such decontamination processes is reduced especially for he lipophilic insecticides, which may penetrate the fruit skin. Accordingly the dislodgable residues were less on the 3rd and 5th day as compared to day 1 sample. Similar results have been reported earlier on another synthetic, pyrethroid cyfluthrin (Sinha and Gopal, 2002).

Overall results indicate that acetamiprid residues can be more effectively dislodged by boiling followed by grilling and frying. Residue reduction was minimal after washing. Besides that, the residues in eggplant fruits and processed products were lower than 0.1 mg kg^{-1} after one day according to the EU MRL (Europe Commission, 2010). This suggests that the use of acetamiprid at the recommended dosage can be considered quite safe from the point of view of health hazards to consumers and a waiting period of 1 day is suggested to reduce the risk before consumption of eggplant fruits and processed products.

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