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Dissipation of Profenofos, Imidacloprid and Penconazole in Tomato Fruits and Products

Ahmed A. Romeh · Tarek M. Mekky · Rady A. Ramadan · Mohamed Y. Hendawi

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Abstract An experiment was conducted to evaluate the effects of some technological processes on the residual levels of profenofos, imidacloprid and penconazole in tomato fruits and products. According to their half-life ($t_{½}$) values, tomato fruits can be safely harvested for human consumption or for processing purposes 3 days after the spray time of imidacloprid and penconazole and 7 days after in the case of profenofos. Pesticide residues were greatly decreased in tomato juice under cold or hot break compared with that taken from unwashed tomato fruits. A sharp decline in profenofos level was noted after treatment by pectinex ultra SP-L and benzyme M during tomato crushing.

Keywords Dissipation · Profenofos · Imidacloprid · Penconazol · Tomato fruits · Tomato products

Pesticides used during agricultural production have increasingly caused concerns because of their adverse effects on human health as they leave residues in varying extents on agricultural produce such as vegetables and fruits. Pesticide residues in/on plants may be unavoidable even when good agricultural practices are used. On the other hand, it was observed that pesticide residues in plant produce are reduced by processing or some household preparation stages such as washing, peeling, cooking, etc. (Dikshit et al. 2003). The effects of some culinary and processing steps such as washing, peeling and cooking on pesticide residues have already been investigated in various fruits and vegetables (Sanyal et al. 2006; Osman et al. 2008). The present investigation aims to study the effects of some technological processes on the residual levels of profenofos, imidacloprid and penconazole in tomato fruits and products.

Materials and Methods

Tomato plants (*Lycopersicon esculentum*) were cultivated in plots consisting of 10 rows separated by a three row belt. Plots were arranged in complete randomized block design at Aboutouala, Mania El-kamh province, Sharkia governorate, Egypt, on 20 March 2008. Common agricultural and fertilization practices were used.

Mature plants were sprayed separately with profenofos 72% EC, imidacloprid 20%SC and penconazole 10% EC at the recommended rates of application- i.e., 540,100 and 10 g ai per feddan (1 feddan = 4,200 m^2) respectively. The amount of formulated pesticide required for 1 feddan was diluted in 400 L of water and applied to plants using a back spray motor. The control plots were left unsprayed. Three replicates were made for each treatment and each pesticide was applied once. The tomato fruits were harvested at a suitable stage maturity, either for consumption or for processing. Fruit samples were taken 1 h after pesticide application to determine the initial deposits of each pesticide. Afterward, the fruits were collected randomly

A. A. Romeh (✉) · R. A. Ramadan
Efficient Productivity Institute Plant Production Department, Zagazig University, Zagazig, Egypt
e-mail: ahmedromeh2006@yahoo.com

T. M. Mekky
Efficient Productivity Institute Food & Dairy Technology, Zagazig University, Zagazig, Egypt

M. Y. Hendawi
Faculty of Agriculture, Plant Protection Department, Zagazig University, Zagazig, Egypt

after 1, 3, 7 and 14 days after application. Random samples of about 500 g were collected from each plot. Different samples of tomato juices were prepared by extraction at room temperature (cold break), at 100°C for 10 min (hot break) or after enzyme treatment.

Some portion of the processed juice was concentrated at 50°C by rotary evaporation (Model rotavapor-R. Buche. Switzerland) until the concentration of the total soluble solids (TSS) reached to 25° brix (measured by refractometer). The hot concentrated paste (25° brix) was transferred into 240 mL colorless bottles and pasteurized at 90°C for 15 min using a boiling water bath.

Representative samples were taken from the treated and untreated tomato fruits; 100 g of the fresh unwashed or washed tomato fruits, 100 mL of the juice and 20 g of the paste. Pesticide residues of the three studied compound were subsequently determined. Representative samples of treated or untreated soil were collected from the surface of the plots to a depth of 10 cm after an hour, 1, 3, 7, and 14 days after treatment. Also samples of tomato green leaves (100 g) were taken at the same periods from corresponding plots. Profenofos and penconazole were extracted from tomato fruits and products according to the method described by Lucke et al. (1975), in leaf samples according to Luke and Doose (1983) and in soil by the method of Krause et al. (1986). Sample extracts were cleaned up as described by Becker and Schug (1990). Subsequently, profenofos and penconazole residues were determined by high-performance liquid chromatography (HPLC). On the other hand, imidacloprid was extracted and cleaned up in tomato leaves, fruits and products according to the method described by Sanyal et al. (2006); while those in soil were extracted using the method described by Shokr et al. (1999). These were cleaned up through deactivated florisil (5%-moisture) using acetonitril as elution solvent then concentrated under vacuum to dryness and the level determined by HPLC. The rate of degradation (K) and half-life ($t_{½}$) values were obtained from the following equation of Gomaa and Belal (1975)

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

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

The residues of the studied pesticides were separated on a stainless steel column (10/250 nm) packed with C18 using a mobile phase of methanol and water (80:20 and 60:40 v/v) for profenofos and imidacloprid at a flow rate of 0.7 and 1.0 mL/min, respectively. For penconazole, a mobile phase of acetonitril and water (80:20v/v) was used at a flow rate of 0.4 mL/min. The column temperature was 40°C in all cases. Quantification was performed against Profenofos, imidacloprid and penconazole standards at a wavelength of 275, 270 and 210 nm, respectively. Under these conditions the retention time of profenofos, imidacloprid and penconazole were 4.38, 3.9 and 6.39 min, respectively. The minimum limits of determination in the soil, leaves, tomato fruits and products were found to be 0.06, 0.03, 0.02 and 0.02 µg/g for profenofos; 0.02, 0.02, 0.01 and 0.01 µg/g for imidacloprid and 0.04, 0.02, 0.01 and 0.01 µg/g for penconazole, respectively. The sensitivity of the method was 002 µg/gm. The control samples of tomato fruits, leaves and soil were spiked at 1.0 µg/g, respectively for each pesticide, and the tomato fruits were processed by the method previously described. Results were corrected according to the percent mean ± SD recoveries (Table 1). Appropriate amounts of each pesticide (profenofos; imidacloprid and penconazole) were added separately to 100 mL distilled water and to 100 g of crushed tomato to bring the final pesticide concentration to 20 ppm. Distilled water and crushed tomato samples were treated with 100 ppm of pectincx ultra SP-L or benzyme-M separately at 45°C for 2 h in crushed tomato and in distilled water for 2

Table 1 Average rate of recovery of Profenofos, imidacoprid and penconazole added to untreated samples at 0.5 ppm

Treatments	Recovery percentages ± SD		
	Profenofos	Imidacoprid	Penconazole
Tomato fruits	85.0 ± 2.5	90.5 ± 2.4	85.8 ± 2.7
Soil	84.6 ± 1.9	91.3 ± 2.2	84.4 ± .7
Leaves	82.00 ± 2.1	85.8 ± 2.4	85.1 ± 4.5
Tomato juice (cold break)	93.3 ± 1.5	93.2 ± 2.5	86.9 ± 2.2
Tomato juice (hot break)	88.4 ± 2.6	90.9 ± 1.3	84.9 ± 3.2
Enzymatic treatment with pectinex ultra SP-L	85.9 ± 1.3	89.8 ± 1.7	84.6 ± 3.0
Enzymatic treatment with benzyme -M	82.40 ± 2.3	86.1 ± 2.0	84.5 ± 2.2
Tomato paste (hot break)	83.5 ± 1.7	84.6 ± 2.2	83.2 ± 3.2
Tomato paste (Enzymatic treatment with pectinex ultra SP-L)	83.7 ± 1.3	84.5 ± 1.8	83.5 ± 3.3
Tomato paste (Enzymatic treatment with benzym- M)	81.3 ± 1.5	83.3 ± 2.9	83.3 ± 2.1

Each value is mean ± SD of three replicates

and 24 h, respectively. Samples of distilled water and crushed tomato treated with pesticides and without the enzymes were considered as the control. Ten ml of water samples from the treated and the control was extracted with methylene chloride for the three pesticides; the crushed tomatoes were extracted and cleaned up as described earlier. After that samples were taken for HPLC determination.

Results and Discussion

The results of the initial deposits, dissipation percentage, and half-life values of the three pesticides in the leaves, soil, and unwashed tomato fruits are presented in Tables 2 and 3. The residues of the three pesticides decreased progressively with time irrespective of application rates. The average initial deposits (1 h after spraying) of profenofos in the soil, leaves and unwashed tomato fruits were found to be 11.71 ± 1.0, 33.95 ± 5.0 and 11.55 ± 2.5 mg/kg, respectively. Fourteen days after application, profenofos was dissipated in the soil and leaves to an undetectable limit; for the unwashed tomato fruits it reached 0.40 ± 0.015 mg/kg. The recorded percentage of dissipation at this period was 100%, 100% and 96.54%, respectively. Likewise, the average initial deposits of imidacloprid in the soil, leaves and unwashed tomato fruits were observed to be 1.39 ± 0.5, 4.22 ± 0.21 and 1.95 ± 0.10 mg/kg, respectively, dissipating into the respective reduced levels of 0.640 ± 015, 0.44 ± 0.04 and 0.075 ± 0.006 mg/kg 14 days after spraying. This indicates considerable rates of pesticide removal amounting to 53.96%, 89.57% and 96.15% of the initial deposits, respectively. Similarly, the corresponding average initial deposits of penconazole in the soil, leaves and unwashed tomato fruits on day 0 (1 h after last spraying) were found to be 0.61 ± 0.015, 1.50 ± 0.26 and 0.24 ± 0.017 mg/kg, respectively. The residue levels decreased to below detectable limit, below detectable limit and 0.048 ± 0.010 mg/kg 14 days after application respectively. The dissipation of pesticide residues in/on crops depends on climatic conditions, type of application, plant species, dosage, interval between application, and time of harvest (Khay et al. 2008). The results also showed half-life ($t_{½}$) values of 27.60, 24.00 and 54.00 h for profenofos; 216.00, 61.92 and 60.00 h for imidacloprid and 72.00, 108.00 and 125.00 h for penconazole in the soil, leaves and unwashed tomato fruits, respectively. The $t_{½}$ of profenofos were found to be 1.84, 1.74 and 1.96 days for hot pepper, sweet pepper and eggplant, respectively (Radwan et al. 2005). As shown in Table 2, it is evident that washing the pesticide-treated tomato fruits with tap water induced considerable relative reductions in pesticide residues within the range of 15.0%–58.75%, 6.67%–30.26% and 6.25%–20.00% for profenofos, imidacloprid and penconazole, respectively. The removal extent depended 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 bound to waxy material in the outer portion of the fruit or vegetable, or translocated into the inner tissues of the plant, with increasing difficulty of removal (Ripley and Edgington 1983). As shown in Table 2, the allowable tolerance level of profenofos, imidacloprid and penconazole in tomato fruits was 2.00, 0.5 and 0.2 mg/kg,respectively as adopted by the FAO/WHO Codex Aliment Arius Commission (CAC 2008). It can thus be concluded that tomato fruits can be safely harvested for human consumption or for processing purposes after 3 days of the spray time of imidacloprid and penconazole whereas, tomato fruits treated with profenofos may be harvested 7 days after last application.

Experimental data (Table 3) on the fate of the pesticide residues during processing were obtained by analyzing samples taken during harvesting, after washing, after juicing and from the tomato paste (the end product). The average amount of profenofos, imidacloprid and penconazole residues in tomato juice under cold and hot break were greatly decreased compared with those in unwashed tomato fruits. Moreover, the amount of residues of the three pesticides was more greatly decreased in tomato juice under hot break than that under cold break. After 3 and 7 days, the reduction percentage of profenofos, imidacloprid and penconazole residues amounted to 72.73%–88.60%, 60.42%–100% and 52.94%–59.23% in tomato juice under cold break while it increased, respectively, to 81.81%–100%, 100%–100% and 68.82%–74.62% under hot break (Table 3). The elevated values of pesticide reduction at hot break could be due to the following; first the heat pretreatment of tomato fruits; second the removal of the fruit rinds in which the pesticide residues were possibly concentrated during straining and third the post-heat preservation treatment (90°C for 15 min) of the pasteurized resulting juice (Ramadan 1990). The residue levels in juices from tomato fruits depend on the partitioning properties of the pesticide between the fruit skin/pulp and the juice. The pulp which often includes the skin retains a substantial proportion of lipophilic residues. Thus moderately to highly lipophilic pesticides are poorly transferred into juices and the residues are further reduced by clarification operations such as centrifugation or filtering (Holland et al. 1994).

The results indicated that enzymatic treatment with pectincx ultra SP-L (100 ppm/45°C/30 min) of unwashed tomato fruits collected after 3–7 days from spraying and subjected to cold break reduced the residues of profenofos, imidacloprid and penconazole by 86.12%–100%, 100%–

Table 2 Dissipation of profenofos, imidacloprid and penconazole in/on leaf, soil and tomato fruits

Days after application	Tomato fruits		Soil		Leaves		Washed tomato fruits	
	Residue level (mg/kg) Mean ± SD	Dissipation %	Residue level (mg/kg) Mean ± SD	Dissipation %	Residue level (mg/kg) Mean ± SD	Dissipation %	Residue level (mg/kg) Mean ± SD	Dissipation %
	Profenofos							
Initial[a]	11.55 ± 2.5	0.00	11.71 ± 1.0	0.00	33.95 ± 5.0	0.00	4.77 ± 0.44	58.7
1	7.91 ± 0.36	31.52	5.94 ± 0.32	49.27	26.06 ± 5.3	23.24	3.47 ± 0.25	56.13
3	4.18 ± 0.20	63.81	0.74 ± 0.021	93.68	4.94 ± 0.40	85.45	2.74 ± 0.46	34.45
7	1.78 ± 0.25	84.59	0.27 ± 0.025	97.69	0.33 ± 0.026	99.03	1.28 ± 0.17	28.09
14	0.4 ± 0.015	96.54	BDL	100	BDL	100	0.34 ± 0.010	15
*t*½ h	54		27.6		24			
RT	0.22×10^{-4} s^{-1}		0.53×10^{-4} s^{-1}		0.44×10^{-4} s^{-1}			
ATL	2.0 mg/kg							
	Imidacloprid							
Initial[a]	1.95 ± 0.1	0.00	1.39 ± 0.5	0.00	4.22 ± 0.21	0.00	1.36 ± 0.6	30.26
1	0.83 ± 0.011	57.43	1.23 ± 0.25	11.51	2.35 ± 0.15	44.31	0.58 ± 0.023	30.12
3	0.48 ± 0.026	75.38	0.95 ± 0.01	31.65	1.38 ± 0.15	67.3	0.38 ± 0.0.01	20.83
7	0.29 ± 0.01	85.13	0.75 ± 0.03	46.04	0.76 ± 0.02	81.99	0.25 ± 0.015	13.79
14	0.075 ± 0.006	96.15	0.64 ± 0.015	53.96	0.44 ± 0.04	89.57	0.07 ± 0.006	6.67
*t*½ h	60		216		61.92			
RT	0.22×10^{-5} s^{-1}		0.13×10^{-5} s^{-1}		0.38×10^{-5} s^{-1}			
ATL	0.5 mg/kg							
	Penconazole							
Initial[a]	0.24 ± 0.017	0.00	0.61 ± 0.015	0.00	1.50 ± 0.26	0.00	0.20 ± 0.0.025	16.67
1	0.20 ± 0.01	16.67	0.44 ± 0.032	27.87	1.22 ± 0.25	18.67	0.17 ± 0.01	15
3	0.17 ± 0.015	29.17	0.23 ± 0.025	62.3	0.94 ± 0.15	37.33	0.15 ± 0.006	11.76
7	0.13 ± 0.01	45.83	0.11 ± 0.005	81.97	0.50 ± 0.02	66.67	0.12 ± 0.0.01	7.69
14	0.048 ± 0.01	80	BDL	100	BDL	100	0.045 ± 0.001	6.25
*t*½ h	125		72		108			
RT	0.04×10^{-5} s^{-1}		0.11×10^{-5} s^{-1}		0.15×10^{-5} s^{-1}			
ATL	0.2 mg/kg							

RT rate of degradation, *ATL* Allowable tolerance level, *BDL* below detectable level

[a] Initial = 1 hour post treatment

100% and 45.29%–56.15%, respectively. When applying benzyme-M instead of pectincx ultra SP-L under the same conditions pesticide reduction levels were increased to higher levels of 97.37%–100%, 100%–100% and 48.24%–61.54%, respectively. It was observed that enzymatic treatment with either pectincx ultra SP-L or benzyme-M (100 ppm/45°C/30 min) was associated with higher retention of penconazole pesticide residue compared with cold break or hot break treatment of tomato juice. This may be due to the liquefaction action by the enzymes (e.g., cellulases and pectinases) on the solid components of the tomato fruit (Pilnik et al. 1989). During liquefaction, cell walls are degraded leading to the release of cell contents which may be recovered with high juice yield and residues (Sreenath and Santhanam 1992). However, the residue levels of profenofos and imidacloprid were more decreased in enzyme-tomato than in tomato juice under cold and hot break. This difference is probably due to the different chemical identities of these pesticides. Comparing the two enzymes, profenofos was more reduced in tomato fruits treated with benzyme-M than in those treated with pectinex ultra SP-L.

Hence, we studied the role of the two enzymes (pectinex ultra SP-L and benzyme M) as applied at 200 ppm/45°C/60 min., in the degradation of the three pesticides studied (profenofos, imdacloprid and penconazole) either as

Table 3 Residues of Profenofos, imidacoprid and penconazole in tomato fruits and products as affected by processing stages

Treatments	Profenofos			Imidacoprid			Penconazole		
	Dying after spraying			Dying after spraying			Dying after spraying		
	1	3	7	1	3	7	1	3	7
Unwashed tomato fruits									
PPM (mean)	7.91	4.18	1.78	0.83	0.48	0.29	0.20	0.17	0.13
±SD	±0. 36	±0.20	±0.25	±0.011	±0.026	±0.010	±0.010	±0.015	±0.010
% Loss	31.52	63.81	84.59	57.43	75.38	85.13	16.67	29.17	45.83
Washed with tap water									
PPM (mean)	3.47	2.74	1.28	0.58	0.38	0.25	0.17	0.15	0.12
±SD	±0.25	±0.46	±0.17	±0.023	±0.006	±0.015	±0.010	±0.006	±0.006
% Loss	56.13	34.45	28.09	30.12	20.83	13.79	15.00	11.76	7.69
Tomato juice (cold break)									
PPM (mean	–	1.14	0.203	–	0.19	BDL	–	0.080	0.053
±SD)	–	±0.010	±0.06	–	±0.01	–	–	±0.010	±0.005
% Loss	–	72.73	88.60	–	60.42	100	–	52.94	59.23
Tomato juice (hot break)									
PPM (mean)	–	0.76	BDL	–	BDL	BDL	–	0.053	0.033
±SD	–	±0.010	–	–	–	–	–	±0.006	±0.006
% Loss	–	81.81	100	–	100	100	–	68.82	74.62
Enzymatic treatment with pectinex ultra SP-L									
PPM (mean)	–	0.58	BDL	–	BDL	BDL	–	0.093	0.057
±SD	–	±0.006	–	–	–	–	–	±0.006	±0.006
% Loss	–	86.12	100	–	100	100	–	45.29	56.15
Enzymatic treatment with benzyme -M									
PPM (mean)	–	0.11	BDL	–	BDL	BDL	–	.088	0.050
±SD	–	±0.006	–	–	–	–	–	±0.006	±0.00
% Loss	–	97.37	100	–	100	100	–	48.24	61.54
Tomato paste (hot break)									
PPM (mean)	–	0.31	BDL	–	BDL	BDL	–	0.087	0.06
± SD	–	±0.006	–	–	–	–	–	±0.006	±0.00
% Loss	–	92.58	100	–	100	100	–	48.82	53.84
Tomato paste (Enzymatic treatment with pectinex ultra SP-L)									
PPM (mean)	–	0.14	BDL	–	0.24	BDL	–	0.102	0.083
±SD	–	±0.006	–	–	±0.006	–	–	±0.005	±0.05
% Loss	–	96.65	100	–	50.00	100	–	40.00	36.15
Tomato paste (Enzymatic treatment with benzym- M)									
PPM (mean)	–	BDL	BDL	–	0.32	BDL	–	0.106	0.086
±SD	–	–	–	–	±0.02	–	–	±0.006	±0.08
% Loss	–	100	100	–	33.33	100	–	37.65	33.85

BDL below detectable level

dissolved in distilled water or in crushed tomato. The data revealed no clear degradation in imidaclopid and penconazole either in distilled water or in crushed tomato. On the other hand, there was a sharp decline in profenofos within 2 h of treatment by the two enzymes in both distilled water and crushed tomato (Fig. 1). This occurred despite the fact that these two enzymes induced liquefaction of the cell walls of tomato fruits and the release of cell contents which may initially enhance the pesticide residues. The overall enzymatic action on pesticide degradation can be explained by the fact that these enzymes include different pectinolytic and cellulolytic, polygalacturonase, endo-pectinylase and pectin esterase as well as other activities, such as _-galactosidase, chitinase and transgalactosidase. The presence of a transfructosylating activity in pectinex ultra SP-L has been also reported (Sohel et al. 2007). These enzymes play a role in

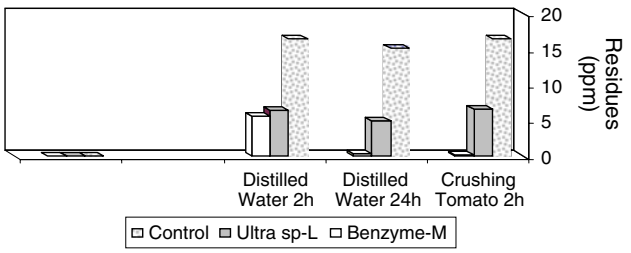

Fig. 1 Dissipation of profenofos under enzymatic treatments

catalyzing some biotransformation reactions (Faber and Kroutil 2005).

In Table 3, it was evident that, when tomato fruits were processed into a paste, the penconazole residues in the paste were concentrated and increased to higher levels than did those in the tomato juice. The relatively high residue levels in juicing by-products can undergo further increases upon drying due to the simple loss of moisture (Holland et al. 1994). This indicates that the concentration process had raised the pesticide residues in the final product. This is quite expected because of the concentration step that transforms the juice into a paste. This was evident despite the possible destructive or reductive effect of both heat treatments carried out to convert tomato juice into tomato paste and the final treatment of the paste, after being packed in the containers (bottles, processed at 90°C for 15 min) for the final preservation (Ramadan 1990). In the same way residues of imidacloprid 3 days after application became undetectable in the tomatoes treated with pectinex ultra SP-L and benzyme M but these were detectable on the tomato paste. In contrast, profenofos residue losses were also apparent when tomato juice was concentrated to paste.

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