PERSISTENCE OF CHLORPYRIFOS-METHYL AND PROFENOFOS RESIDUES IN FRESH AND PROCESSED TOMATO FRUITS

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INTRODUCTION

Tomato, *Lycopersicon esculentum* is considered an important vegetable crop in Egypt. Tomato plants are infested by many pests such as the aphid, *Aphis gossypii*, the whitefly, *Bemisia tabaci*, the Egyptian cotton leaf worm, *Spedoptera littoralis*, the potato tuber worm, *Phthorimaea operculella* and the American bollworm, *Heliothis armigera* (Anonyomus, 2001). Both chlorpyrifos-methyl and profenofos are used in controlling these insects. Using insecticides could contaminate tomato fruits and cause hazard to consumers. Many publications revealed the existence of pesticide residues, mainly organophosphorus insecticides in tomato fruits. (El-Sayed, *et al.*, 1971; Gomaa, *et al.*1985; Ramadan 1991; El-Nabarawy ,*et al.*1992; Abdalla, *et al.*,1993 and Sallam1998).

Analysis of pesticide residues after application to vegetable plants should be followed to determine the safety period between application and harvesting to be sure that residues are below tolerance levels and the edible parts are safe for human consumption (El-Sayed *et al.*, 1971; and Shokr, 1997). Many studies were carried out on the removal of pesticide residues from various vegetables (Fahey, *et al.*, 1969 & 1971; Tantawy, *et al.*, 1975; Mesallam and Moharram 1980; Ramadan, 1991; Ismail, *et al.*, 1993; Zidan and El-Daim, 1996 and Zidan *et al.*, 1997)

This work aimed to study the persistence of the aforementioned insecticide residues in tomato fruits; to give an idea about the pre-harvest interval (PHI) that should pass following application and before marketing in order to minimize health hazards. This study also aimed to throw light on the effect of washing methods and processing steps on the removal of insecticide residues from tomato fruits.

MATERIAL AND METHODS

Insecticides used are: a-Chlorpyrifos-methyl: O, O, dimethyl, O-(3, 5, 6-trichloro-2-pyridinyl) phosphorothioate. The formulation Reldan 50 % E.C. was used at the rate of (250 cm^3/ 100 liters of water). b-Profenofos: O-(4-bromo-2-chlorophenyl)–O,ethyl-S-propylphosphorothioate (Anonymous, 1993). The formulation Selecron 72 % E.C., was used at the rate of (187.5 cm^3/100 liters of water), using 200 liters of water per feddan applied by a knapsack sprayer.

The field experiment was carried out in the experimental Farm of the Faculty of Agriculture, Sohag and South Valley University. Tomato *Lycopersicon esculentum* Var., Castlerock seedling was cultivated during the summer cultivation season of 2004, under normal field and agricultural practices. The experimental area was divided into plots of 42 m^2 ($^1/_{100}$ fed.). The experimental area design was a complete randomized blocks with three replicates for each insecticide. Three plots were left untreated to serve as control. The insecticide applications were carried out on May, 10, 2004 at the rates mentioned above using a knapsack sprayer.

Representative samples of tomato fruits were collected from the treated plots after one hour of application (initial deposits) and 1, 3, 5,7,9,12,15 and18 days after spraying. Clean polyethylene bags were used for preserving the collected samples. The samples were stored at – 20 °C in a deep freezer until analysis.

Different methods of washing were tested:

1-Washing with tap water: Tomato fruits were washed with running water for five minutes.

2-Soaking and washing: tomato fruits were soaked for ten minutes in tap water, followed by washing with tap water for five minutes.

3-Washing with synthetic detergent (Ariel): This detergent powder contains (anionic surfactant and phosphate): Tomato fruits were washed by 1 % detergent solution for five minutes, followed by washing with running water for five minutes.

4-Washiing with white vinegar (acetic acid 5%): Tomato fruits were soaked for five minutes in vinegar solution (1 %), followed by washing with running water for five minutes. All samples after washing were left to air dry and kept in polyethylene bags in a deep freezer until analysis.

Samples of tomato juice were prepared after washing fruits with tap water as mentioned above. The washed fruits were transferred to hot cooking kettle, which was heat controlled at 90°C and the heating process was continued for one minute to minimize any undesirable changes taking place under otherwise conditions. Tomato juice was then obtained by crushing tomato fruits in a strainer and receiving the juice in other kettle free from seeds and remnants.

Tomato paste was prepared from tomato juice which was transferred to special kettle provided with a heating system for the concentration of the total soluble solids up to 25 % In addition, 2.5 % NaCl was added to tomato concentrate during the process of concentration.

Residue analysis:

The frozen samples were left to reach room temperature. Fifty grams of samples were placed in the blender cup with 50 g. anhydrous sodium sulphate and 150 ml. ethyl acetate, and then blended for five minutes. The liquids were decanted through a funnel with a plug of cotton into a graduated cylinder then evaporated just to dryness using a rotary evaporator at 40 °C. (Anonymous, 1985). The residues were dissolved in 5 ml of n- hexane and clean up was done according to Mills, *et al.* (1972).Through chromatography column 10 g. of activated florisil 60-100 mesh 3.5% moisture covered with 2g. of anhydrous sodium sulphate. The elution solvent system was dichloromethane: n- hexane: acetonitrile at the ratio of 50: 48.5: 1.5.The elute was then collected in a 250 ml flask evaporated under vacuum to dryness. Residues were redissolved in the suitable volume of ethyl acetate for GC analysis.

A Pyeunicam 4500 gas chromatograph equipped with FPD operated in the phosphorous mode and a Pyrex glass column (1.5 × 4mm i.d.) packed with 4 % SE-30 + 6 % OV-230 on gas chromosorb Q 80-100 mesh was used under the following conditions: Injector temperature 240 °C, Column temperature 240 °C, Detector temperature 250°C, Carrier gas (N_2) flow rate 4 ml/min., hydrogen and air flow rate: 8&100 ml/min., respectively. Chlorpyrifos-methyl and profenofos retention time under these condition were 2.48 and 4.24 minutes, respectively.

Untreated tomato fruits were fortified by the addition of standard solution of chlorpyrifos-methyl and profenofos at levels ranged from 0.1 to 1.0 ppm. The fortified samples were processed through all steps of the analytical method to validate the assay procedure. Results were corrected according to the recovery percentages obtained from fortified untreated samples (Table 1).

RESULTS AND DUSCUSION

Recovery percentages of chlorpyrifos-methyl and profenofos:

Recoveries of chlorpyrifos-methyl and profenofos from fortified samples (50 g.) at different stages in processing are shows in Table (1). Recoveries ranged from 90.5 to 93.5 % for chlorpyrifos-methyl, whereas profenofos was 88.7 to 92.8 %. The average of recoveries for chlorpyrifos-methyl with unwashed tomatoes; 93.5 % washed tomatoes 92.2 % tomato juice and tomato paste 90.5 %.The corresponding values of profenofos were: 90.6.; 92.8.; 89.8 and 88.7 %. These results agree with those obtained by Hegazy, *et al.* (1997) using the same method of analysis of chlorpyrifos-methyl residues on cucumber. Rate of recovery was 100 %. Sallam and El-Nabarawy (2001) using also the same method in determination of chlorpyrifos-methyl, chlorpyrifos, and profenofos on moloukhia leaves. They found that recovery percentage of these insecticide were 86.52, 90.43, and 83.4 %, respectively.

TABLE (I)

Recovery percentages of chlorpyrifos-methyl and profenofos from tomato fruits

Added (ppm)	Chlorpyrifos-methyl				Profenofos			
	Unwashed	Washed	Juice	Paste	Unwashed	Washed	Juices	Paste
1.0	94.5	94.0	93.0	92.5	91.4	94.4	90.5	91.0
0.5	93.0	93.6	92.0	90.0	90.4	92.6	90.0	87.0
0.1	92.5	93.0	91.6	89.0	90.0	91.5	88.8	88.1
Average	93.3	93.5	92.2	90.5	90.6	92.8	89.8	88.7

Persistence of chlorpyrifos-methyl and profenofos on and in tomato fruits:

Results in Table (2) show that the concentration of the initial deposits of chlorpyrifos-methyl and profenofos on unwashed tomato fruits were 2.6400 and 3.4696 ppm., respectively. The amount of residues decreased to 1.2400 and 2.0781 ppm., respectively within the first 24 hours after spraying .The residues of chlorpyrifos-methyl dropped to o.5120, 0.1240, 0.0384,0.0192,0.0015, and undetectable after 3,5,7,9,12,and 15 days, respectively. The corresponding values for profenofos were 1.6430, 1.1330, 0.7080, 0.3665, 0.2235 and 0.0969 ppm.

The results agree with that of Abdalla, *et al.* (1993), Badawy, *et al.* (1995) and Abd El-Daim and Zidan, (1996). They found that the initial deposits of

profenofos in unwashed tomato fruits after one hour of application were 2.85, 3.42 and 2.14 ppm, respectively.

The amount of residues recorded during the experimental period varied for each insecticide to another. These levels depended on the initial deposits, the rate of exposure of the fruits to the environmental factors and the reaction between the treated surface and the chemical applied. Stevens, *et al.* (1988) demonstrated that uptake of pesticides on plant surface is affected by, the chemical structure, formulation as well as the rate of used insecticide, the nature of the recipient surface, the used spraying equipment and the climatic conditions; especially the ambient temperature during pesticide application.

Data presented in Table (2) show that loss of residues increased as time lapsed from the onset of spraying until the end of the experimental period. After one day from the onset of spraying loss percentages were 53.03 and 40.11, with chlorpyrifos-methyl and profenofos, respectively .After the 5th day more than 90 % of chlorpyrifos-methyl residues were disappeared. While with profenofos ,more than 60 % of the initial deposits were disappeared .The same phenomenon was observed by Marei *et al.*(1979).They found that loss percentages of chlorpyrifos was 100 % after 9 days when exposed to sunlight in open beakers. Shiboob (1995) also found that loss percentages of profenofos residues in tomato and cucumber fruits ranged from 99.1 to 99.3 % after 12 days from spraying.

TABLE (II)

Residues (ppm) of chlorpyrifos-methyl and profenofos at indicated period of spraying in and on tomato fruits

Days after spraying	Chlorpyrifos-methyl		Profenofos	
	Unwashed	% dissipation	Unwashed	% dissipation
One hour *	2.6400	00.00	3.4696	00.00
1	1.2400	53.03	2.0781	40.11
3	0.5120	80.61	1.6430	52.65
5	0.1240	95.03	1.1330	67.34
7	0.0384	98.55	0.7080	79.59
9	0.0192	99.27	0.3665	89.44
12	0.0015	99.94	0.2235	93.56
15	UN **	100.00	0.0969	97.21
18	UN	100.00	0.0671	99.07

* Initial deposits of the insecticide. ** Undetectable

The values of half-life were obtained from calculations of Moye, *et al.*, (1987). The half-life periods of chlorpyrifos-methyl and profenofos residues in unwashed tomato fruits were 1.23 and 2.57 days, respectively. This results are in agreement with those obtained by Abdalla,*et al.*(1993) who found that the half-life value (RL_{50}) of profenofos residues was 3.2 days on tomato fruits, while with Sallam (1998) was 2.1days. On other hand, the present results differed from those obtained by Ahmed and Moursy (1991) who mentioned that profenofos residues had persisted in garlic, tomato, and strawberries for up to three weeks after the second foliar application of profenofos. Hegazy *et al.*(1997) found that half-life value of chlorpyrifos-methyl residues on cucumber fruits was 17 hours .Sallam& El-Nabarawy (2001) stated that the half-life values of chlorpyrifos-methyl ,chlorpyrifos and profenofos on moloukhia leaves were 22.8,33.84,and 52.08 hours, respectively.

It is obvious that chlorpyrifos-methyl was degraded faster than profenofos (Fig.1). These differences in the rate of disappearance of this insecticide may be attributed to the differences between chemical structure and formulation as well as the rate of application. The same conclusion was pointed out by El-Tantawy and Hussein (1978) who mentioned that degradation rate was correlated to the chemical structure of the tested compounds.

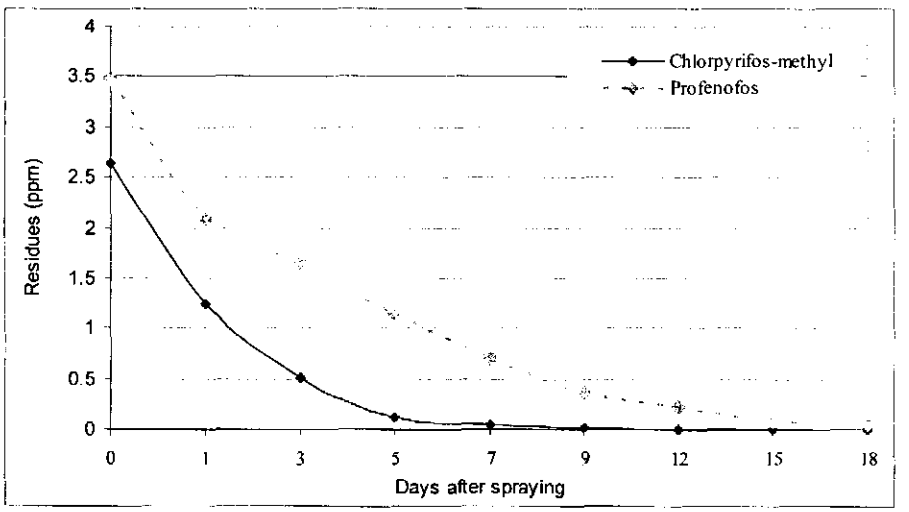

Fig (1): Persistence of chlorpyrifos-methyl and profenofos residues in and on tomato fruit

According to the maximum residues limits (MRL_s) of chlorpyrifos-methyl (0.5 ppm), and profenofos (2 ppm) in tomato fruits, presented in Anonymous (1997), chlorpyrifos-methyl and profenofos–sprayed tomato fruits can be picked up after 5

and 3 days, respectively from spraying. The same conclusion was pointed out by El-Sayed, *et al.* (1977) They reported that waiting periods between application of insecticides and harvesting for marketing were defined for the consumer safety and avoiding health hazards, ranged between one and twelve days according to kinds of pesticides and vegetables .Hegazy, *et al.*, (1997) indicated that only three days period was enough for the chlorpyrifos-methyl residues in cucumber to reach a safe level less than the (MRLs) (0.5ppm).

Effect of washing in removing chlorpyrifos-methyl and profenofos residues:

Data in Table (3) indicate the great influence of washing with different methods in removing or elimination of chlorpyrifos-methyl and profenofos residues from sprayed tomatoes. After one day of spraying, the residues of chlorpyrifos-methyl on unwashed tomato fruits were 1.2400 ppm. These were reduced to 0.6060, 0.4807, 0.0402 and 0.0086 ppm on tomato washed with tap water, soak-rinse washed, washed with detergent (Ariel) and washed with white vinegar (acidic acid 5%). The corresponding figures for profenofos were 2.0781; 0.9926; 0.7482; 0.2748; and 0.0249 ppm. Washing process removed residues from 51.22 to 99.31 and 52.24 to 98.80 % for chlorpyrifos-methyl and profenofos on tomato fruits. Such findings are in agreement with those obtained by Fahey *et al.* (1969 & 1971), Tantawy *et al.* (1975), Kamil (1987), Ramadan (1991), Badawy , *et al.* (1995) Abd El-Daim and Zidan (1996), Zidan, *et al.* (1997) and Sallam (1998)

TABLE (III)

Effect of processing tomato fruits on the elimination of chlorpyrifos-methyl and profenofos insecticide residues after one day of spraying

Processing steps	Chlorpyrifos-methyl		Profenofos	
	Residues (ppm)	% Dissipation	Residues (ppm)	% Dissipation
Unwashed	1.2400	00.00	2.0781	00.00
Washed with tap water	0.6060	51.22	0.9926	52.24
Soak-rinse washed	0.4807	61.23	0.7482	64..00
Washed with detergent (ariel)	0.0402	96.76	0.2748	86.78
Washed with white vinegar	0.00086	99.31	0.0249	98.80
Juicing	0.1644	86.74	0.2925	85.92
Pasting	0.0036	99.71	0.0066	99.68

As a general trend, washing process caused considerable removal of chlorpyrifos-methyl and profenofos residues. This may be attributed to hydrolysis of the esters, in addition to mechanical effect (Wen, *et al.*, 1985)

It is generally observed from results obtained that white vinegar and detergent (ariel) treatment gave the highest percent removal of the insecticides from tomato fruits. Finally it can be concluded that using vinegar (acetic acid 5 %) and detergent are recommended as economical and practical washing treatment with satisfactory residues.

Residues in tomato juice and paste:

Data in Table (3) concerning the role of technological processes in removing chlorpyrifos-methyl and profenofos residues from contaminated tomatoes. The juice made from washed tomatoes contained from 0.1644 and0.2925 ppm of chlorpyrifos-methyl and profenofos residues, respectively .The corresponding values in tomato paste were 0.0036 and 0.0066 ppm. Juice extraction caused removal percentages of 86.74 and 85.92 % for chlorpyrifos-methyl and profenofos residues, while paste concentration removed nearly all insecticide residues (99.71 and 99.68). This finding are in harmony with those of Kamil (1987) who found that tomato juicing led to a reduction of 92.96 and 98.99 % of dimethoate and actellic. El-Nabarawy *et al.* (1992) stated that the residues of profenofos and malathion in tomato paste were 0.065 and 0.558 ppm, respectively. Abd El-Daim & Zidan (1996) found that insecticide residues on tomato completely removed by one of six sequential processes. Tomato paste removed all insecticide residues. Generally, removal of residues by cooking process may be attributed to decomposition of residues by heating in addition to hydrolysis in water

It is finally observed from this study that tomato fruits could be safely consumed after processing steps that remove most insecticide residues to below the (MRLs).

SUMMARY

Tomato plants were sprayed with chlorpyrifos-methyl (Reldan 50 % E.C.) and profenofos (Selecron 72 % E.C.), at the rate of (250 and 269 g.a.i./fed.),respectively. Persistence of these insecticide residues on and in tomato fruits was studied. This study also aimed to throw light on the effect of processing steps on the removal or elimination of these residues from tomato fruits .The results showed that the amount of residues recorded during the experimental period, varied for each insecticide to another .The initial deposits of chlorpyrifos-methyl and

profenofos on and in unwashed tomato fruits were 2.6400 and 3.4696 ppm, respectively. These figures were decreased to 0.0015 and 0.2235 ppm after 12 days of spraying. The first three days were, however, the most critical period at which most amounts of residues were dissipated. Chlorpyrifos-methyl was degraded faster than profenofos (the half-live values were 1.23 and 2.57 days, respectively).

According to maximum residues limits (MRLs) the safety period after which sprayed tomato fruits can be picked up were 5 and 3 days, respectively.

Data also indicated that all processing steps caused a significant reduction in insecticide residues and varied in their efficiency of removing or elimination. Washing process with different methods removed amounts ranged from 51.22 to 99.31 % and 52.24 to 98.80 % of chlorpyrifos-methyl and profenofos residues on tomato fruits. Tomato juice and paste led to a reduction of 86.74 and 99.71%.; 85.92 and 99.68 % of chlorpyrifos-methyl and profenofos residues, respectively. Finally it may be concluded that tomato fruits could be safely consumed after processing, as most insecticide residues were found below the (MRLs).

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