RESIDUES AND HALF-LIVES OF FENITROTHION AND PIRIMIPHOS-METHYL IN FRUITS OF CUCUMBER AND PEPPER PLANTS GROWN IN GREENHOUSES

BY

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ABSTRACT

Cucumber, sweet pepper and hot pepper plants grown in an experimental plastic house were sprayed with fenitrothion (Sumithion 50% EC) and pirimiphos-methyl (Actellic 50% EC) at their recommended rates. Initial deposits of fenitrothion and pirimiphos-methyl on and in cucumber, sweet pepper and hot pepper fruits were 2.32 and 9.14 ppm, 4.46 and 10.16 ppm and 12.11 and 12.16 ppm, respectively. Under greenhouse conditions fenitrothion and pirimiphos-methyl had shorter residue half life values (RL_{50}) which were 14.4 and 14.4 hours, 22 and 21.6 hours, and 18 and 19.2 hours on cucumber, sweet pepper and hot pepper fruits, respectively.

In this experiment the recommended waiting time values (preharvest intervals) of fenitrothion and pirimiphos-methyl were 3 and 2 days, 7 and 4 days and 7.8 and 7 days for cucumber, sweet pepper and hot pepper, respectively. Residues of fenitrothion and pirimiphos-methyl on these vegetable fruits, 8 days after spraying were very much less than the codex MRLs, also the waiting time values for fenitrothion were longer than the corresponding values for pirimiphos-methyl on these vegetable fruits.

The removal percent after washing process were removed 15.38, 78.24, 65.65, and 25.45, 77.40, 31.13 of fenitrothion and pirimiphos-methyl from cucumber, sweet pepper and hot pepper fruits, respectively. The removal percent of both pesticides from sweet pepper fruits were higher than from cucumber and hot pepper fruits. Also the washing process after one day of spraying did not have efficiency to remove residues of fenitrothion and pirimiphos-methyl to a level below the codex MRLs.

Key words: Residues, Fenitrothion, pirimiphos-methyl, cucumber, pepper.

MATERIALS AND METHODS

Cucumber (Primo) F_1-Hybrid, sweet pepper (Tarek) F_1-Hybrid and hot pepper (Pical) F_1-Hybrid plants were grown in the greenhouses of Ministry of Agriculture located in Dokki-Giza Region, Egypt. Greenhouse temperatures ranged between 24 to 31°C during May 2003.

The plants at fruiting stage were sprayed with Sumithion (50% EC) and Actellic (50% EC) on May 7th 2003, at rates 250 and 375 ml from the commercial products, each per 100 L of water (recommended dose), respectively, using motorized sprayer. Replicate samples, one Kg. each, of cucumber and pepper fruits were taken at intervals of one hour after application (zero time), 1, 3, 5 and 8 days.

As soon as the fruits were picked up, they were put in polyethylene bags and transferred to the laboratory. The fruits were chopped and blended. The blending was thoroughly mixed, three sub-samples 100 g. of each were weighted into 200 ml beakers, capped with aluminum foil and kept deep frozen until analysis.

After one day of application another equal sample of each fruits were rinsed for three minutes with running tap water, then drained on a clean paper for one hour until dry and processed as mentioned before.

Analytical procedures.

A. Extraction:- The method of Mollhoff (1975) was adopted for extraction of the fenitrothion and pirimiphos-methyl from cucumber and pepper fruits, methanol was used instead of acetone. One hundred gram sample of fruit was placed in the blender cup and a constant amount of methanol (2 ml/gram fruit) was added, then blended for three minutes and filtered. Extracts were shaken successfully with 70, 50 ml and 50 ml of methylene chloride in separatory funnel after adding 40 ml of sodium chloride solution (20%); then the water phase was discarded. The combined methylene chloride phases were dried by filtration through anhydrous sodium sulphate. Then, it was evaporated just to dryness using a rotary evaporator at 40°C.

B. Clean-up of Extracts:- The clean-up procedure done according to the method of Mills *et. al.* (1972). An elution solvent system used a mixture of 50% methylene chloride – 1.5% acetonitrile – 48.5% hexane (v/v/v). A column chromatographic containing 10 g.

activated florisil for fenitrothion and pirimiphos-methyl extract was used. Then the residues from the column were eluted with 200 ml of this mixture. The eluant was evaporated just to dryness as previously described and the residues were ready for chromatographic determination after being redissolved in an appropriate volume of ethyl acetate.

C. Gas Liquid Chromatography Determination:- GC-systems – HP 6890 Series equipped with a flame photometric detector operated in the phosphorus mode (526 nm filter) was used to determine fenitrothion and pirimiphos-methyl. The capillary column PAS-1701, 25 m x 0.32 mm, with 0.25 μm film thickness of (14% - cyanopropyl-phenyl) - methylpolysiloxane. Operating conditions: nitrogen carrier gas 3 ml/min, hydrogen 75 ml/min, air 100 ml/min and make up nitrogen 12 ml/min. Temperature degrees were 250°C for detector, 240°C for injector and initial temperature 200°C, initial time 2 min, rise 5°C/min and final temperature 240°C for oven. Hold time 10 min.

Results were corrected according to the rates of recovery which were determined in fortified untreated samples at levels ranged from 0.1 ppm to 1 ppm. Following the techniques previously mentioned, the rates of recovery for fenitrothion and pirimiphos-methyl were 95.21%, 96.86% and 97.59%, 98.47% in cucumber and pepper fruits, respectively.

RESULTS AND DISCUSSION

The data obtained in Table (1) show that the maximum concentrations (initial deposits) of fenitrothion and pirimiphos-methyl on and in cucumber, sweet pepper and hot pepper fruits, one hour after application were 2.32 and 9.14 ppm, 4.46 and 10.16 ppm, and 12.11 and 12.16 ppm, respectively. The different levels of initial deposits of both tested pesticides on fruits of cucumber, sweet pepper and hot pepper mainly due to many factors; the ratio of surface to mass area and character of treated surface (smooth or rough and waxy or non-waxy) (Abo El-Ghar and Ramadan, 1962); systemic and non-systemic character of both compounds, high wax

Table (1). Residues of fenitrothion and pirimiphos-methyl on and in some vegetable fruits in greenhouses.

Time after application (days)	Residues (ppm)					
	Cucumber fruits		Sweet pepper fruits		Hot pepper fruits	
	Fenitrothion	Pirimiphos-methyl	Fenitrothion	Pirimiphos-methyl	Fenitrothion	Pirimiphos-methyl
zero time*	2.32	9.14	4.46	10.16	12.11	12.61
1	0.39	1.65	1.93	4.69	3.93	4.53
3	0.019	0.18	0.55	2.06	0.68	3.33
5	0.002	0.03	0.32	0.49	0.51	2.91
8	N.D**	0.005	0.01	0.05	0.075	0.12
RL_{50} in hours	14.4	14.4	22	21.6	18	19.2
MRL (ppm)	0.05	1	0.1	1	0.1	1
Approximate waiting periods(days)	3	2	7	4	7.8	7

* One hour after application.
** Not detectable.

content of fruit surface and hydrophilic-Lipophilic balance of investigated pesticides controlled the penetrability of applied agrochemicals into fruit tissues (Polen, 1971 and Cabras *et. al.*, 1988).

On the other hand the differences in initial retention of fenitrothion and pirimiphos-methyl between fruits of cucumber, sweet pepper, and hot pepper probably relates to their different surface to weight ratios and perhaps, different surface properties. The present study confirmed that initial deposits of pirimiphos-methyl on and in cucumber and sweet pepper fruits (9.14 and 10.16 ppm) were higher than the initial concentrations of fenitrothion (2.32 and 4.46 ppm). Such difference could be attributed to the higher rate of application of pirimiphos-methyl 350 ml (i.e. 175 g a.i.)/100 L water than fenitrothion 250 ml (i.e. 125 g a.i.)/100 L water. The level of the initial residue of pesticides is determined by many factors i.e. applied dosage, meteorological factor and biological factors depend on the kind of the plant surface.

A rapid loss of fenitrothion and pirimiphos-methyl residues on these vegetables were observed within one day following application, which is approximately ranged between 54% to 83%. Volatilization appears to be the major factor in this loss (Vettorazzi, 1976). The initial deposits of pesticides are influenced by different factors: evaporation of the surface residue which is dependent on temperature condition, biological dilution which is dependent on the increase mass of fruits or enlargement of surface leaves, chemical or biochemical decomposition, metabolism and photolysis. Between days 1 and 8 after spraying a gradual decrease was observed in residues for fenitrothion and pirimiphos-methyl which reached N.D and 0.005 ppm, 0.01 and 0.05 ppm, and 0.075 and 0.12 ppm on and in cucumber, sweet pepper and hot pepper fruits, 8 days after treatments, respectively.

The fast disappearance of fenitrothion and pirimiphos-methyl may be due to its higher vapor pressure (15 mPa at 20^{o}C) for fenitrothion and (15 mPa at 30^{o}C) for pirimiphos-methyl (these values according to Tomlin, 1997), and also other factors such as weathering, metabolic conversions or other degradation processes. However, the first day following application is critical in the sense sharply decreases which are approximately ranged between 54% to 83% from the initial deposit. These results agreed with the findings of Gruzdyev *et al.* (1983) who reported that after the treatment of plants with pirimiphos-methyl was rapidly (in two or three days) vanishes from their surface because of evaporation. Under other conditions such as lower application rate or under field conditions, considering that pirimiphos-methyl loss from plant surface is mainly by volatilization (FAO/WHO, 1983) which may reduce initial retention and half-life, waiting period may be acceptable. Robertson (1977) stated that pirimiphos-methyl is translaminar and has a short persistence in growing tissue. When fenitrothion gets onto a plant, rapidly penetrate into its tissues (over 50% of the total amounts in 24 hours), but does not migrate along the vascular system, exhibiting only a penetrating effect, it decomposes in a plant quit actively in three main directions: hydrolysis to dimethyl phosphorothioic acid and 3-methyl-4-nitrophenol with the following conjugation of the latter with glucuronic and sulphuric acids or glucose, *0*-demethyltion; and oxidation to the oxy analogue (Sumioxon) with subsequent *0*-demethyltion or hydrolysis (Gruzdyev *et al.*, 1983).

In this experiment under greenhouse conditions, fenitrothion and pirimiphos-methyl had shorter residue half-life values (RL_{50}) which were 14.4 and 14.4 hours, 22 and 21.6 hours, and 18 and 19.2 hours on cucumber, sweet pepper, and hot pepper fruits, respectively. These results are in agreement with those of Shokr (1997) who studied the residues of fenitrothion and pirimiphos-methyl on and in some vegetable crops and found that the half-live values of these insecticides were 10.6, 26.1 and 8.2 hours and 11.3, 27.8 and 15.9 hours on moloukhia leaves, green beans pods and cucumber fruits, respectively. Similar results were reported by Abd-Alla *et al.* (1994) who found that half-life value of pirimiphos-methyl was 0.6 days in unwashed tomato fruits. Abdel-Rahman (1996) found that fenitrothion showed short persistence as the half-life period in garlic heads was 1.1 days. Shokr *et al.* (2000) reported that the half-live values of pirimiphos-methyl were 15.6 and 15.6 hours on green pods and peels of broad bean plant. The half-life of pirimiphos-methyl was 1.4 days in whole cawpea pods (Barakat *et al.*, 1994). Under greenhouse conditions, pirimiphos-methyl had a shorter half-life than the other insecticides (Antonious and Snyder, 1994). Generally it was found that organophosphorus pesticides persist for short periods in plants and other environmental constituents (Edwards, 1973).

According to the Codex Alimentarius Commission (1990), the maximum residue levels (MRLs) for fenitrothion and pirimiphos-methyl on cucumber, sweet pepper and hot pepper fruits were 0.05 and 1 ppm, 0.1 and 1 ppm, and 0.1 and 1 ppm, and recommended approximate waiting time values (preharvest intervals) were 3 and 2 days, 7 and 4 days, and 7.8 and 7 days, respectively. Slight amount of fenitrothion and pirimiphos-methyl on and in these vegetable fruits, 8 days after spraying were too much below the permissible residue limits. Also the waiting time values for fenitrothion were longer than the corresponding values for pirimiphos-methyl on these vegetable fruits, this result could be attributed to the maximum residues limits (MRLs) for pirimiphos-methyl (1 ppm) higher than the maximum residues limits (MRLs) for fenitrothion (0.5 and 0.1 ppm) on these vegetable fruits. The low persistence of pirimiphos-methyl on vegetation and the low no-effect levels permitted in food for human consumption allows pirimiphos-methyl to be applied shortly before harvest of edible crops (Robertson, 1977). The low mammalian toxicity, lack of toxic metabolites, and its efficiency against insects as a broad spectrum, contact insecticide on vegetables and fruits (Tomson, 1979) indicate that pirimiphos-methyl may be a potential substitute for other, more toxic organophosphorus insecticides. Consequently insecticides having long post-application waiting periods are not compatible with vegetable production, especially during fruit ripening (Antonious and Snyder, 1994). The safe period of harvesting vegetables treated with the organophosphorus insecticides, ranged between 1 and 12 days post treatment, depending on the chemistry of tested pesticide and kind of crop (El-Sayed *et al.*, 1977 and Khan *et al.*, 1985).

Several investigators had studied the degradation behaviour of pesticides in greenhouse vegetable fruits, Shahin *et. al.* (1989-a and-b) found that the initial deposits of Sumithion were 1.90 and 3.59 ppm on and in greenhouse cucumber and pepper fruits, pesticide undetectable on and in cucumber fruits 9 days treatment, but was 0.0005 ppm on and in pepper fruits 10 days after application. Residues of fenitrothion on cucumber fruits in protected house decreased from 0.32 to 0.01 ppm, 10 days after spraying, also fruits could be consumed safely 6 days after application (Al-Azawi *et. al.*, 1991). Initial deposition of pirimiphos-methyl and its disappearance rate on the different types of plant surfaces in greenhouse varied widely, while residue disappearance rates varied from rapid on cucumber fruits ($t_{1/2}$ = 1.8 days) to slow on pepper fruits ($t_{1/2}$ = 4.3 days) over 42-day period (Antonious and Snyder, 1995).

Cucumber and pepper fruits before being used for any purpose whether for direct consumption or even for further processing are mostly washed before use probably to remove dust and adhering substances rather than removing pesticide residues. The results in Table (2) showed the residues levels in ppm and the percent of removal of each tested pesticide after washing with tap water. The residues of fenitrothion and pirimiphos-methyl on unprocessed cucumber, sweet pepper and hot pepper fruits, after one day from spraying with these pesticides were 0.39, 1.93 and 3.93 ppm and 1.65, 4.69 and 4.53 ppm, respectively. This process reduced these residues to 0.33, 0.42 and 1.35 ppm and 1.23, 1.06 and 3.12 ppm for fenitrothion and pirimiphos-methyl on cucumber, sweet pepper and hot pepper fruits, respectively, with corresponding percent of removal of 15.38, 78.24 and 65.65% and 25.45, 77.40 and 31.13%, respectively. The percent removal of both pesticides from sweet pepper fruits were higher than from cucumber and hot pepper fruits. In this experiment observed the washing process did not have efficiency to remove residues of fenitrothion and pirimiphos-methyl to a level below the codex MRLs, this may be attributed to adhesion of these residues on vegetable fruits.

According to data of Table (2) and remakes of Polen (1971) and Elkins (1989), removal of pesticide residues by washing depends on several factors: character of the surface of the plant foods (smooth or rough, waxy or non-waxy); surface to volume ratio (washing is effective for bigger fruits); reference point of residue levels (higher levels easier to remove); chemical and physical properties of the applied pesticides; the length of time that the pesticide has been in contact with the plant foods; rate and number of applications and penetrability of pesticide into fruit tissues. Also, the washing processes were found to be efficient in removing organophosphorus pesticides from vegetables (Shahin *et. al.*, 1989-a and-b; Al-Azawi *et. al.* 1991; and Shokr 1997).

Table (2). Effect of washing process on fenitrothion and pirimiphos-methyl residues in some vegetable fruits, after one day of spraying in greenhouses.

Crop	Process	Fenitrothion		Pirimiphos-methyl	
		ppm	% removal	ppm	% removal
Cucumber fruits	Unprocessed*	0.39	00.00	1.65	00.00
	Washing	0.33	15.38	1.23	25.45
Sweet pepper fruits	Unprocessed*	1.93	00.00	4.69	00.00
	Washing	0.42	78.24	1.06	77.40
Hot pepper fruits	Unprocessed*	3.93	00.00	4.53	00.00
	Washing	1.35	65.65	3.12	31.13

* After one day from application.

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الملخص العربى

متبقيات وفترات نصف العمر لمبيدى الفنتروثيون والبيريموفوس- ميثيل فى ثمار نباتات الخيار والفلفل المنزرعة فى الصوب الزجاجية

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عوملت نباتات الخيار والفلفل الحلو والفلفل الحار المنزرعة فى البيوت المحمية بمبيدى الفنتروثيون (سوميثيون ٥٠% مركز قابل للاستحلاب) والبيريموفوس- ميثيل (أكتيليك ٥٠% مركز قابل للاستحلاب) بالمعدلات الموصى بها. حيث كانت كميات المتبقى الأولى للمبيدين على وفى ثمار الخيار والفلفل الحلو والفلفل الحار كالتالى ٢,٢٣ ، ٩,١٤ جزء فى المليون و ٤,٤٦ ، ١٠,١٦ جزء فى المليون و ١٢,١١ ، ١٢,٦٠ جزء فى المليون على التوالى . أوضحت الدراسة أن فترات نصف العمرلمتبقيات الفنتروثيون و البيريموفوس- ميثيل قصيرة حيث كانت ١٤,٤ ، ١٤,٤ ساعه و ٢٢ ، ٢١,٦ ساعه و ١٨ ، ١٩,٢ ساعه على ثمار الخيار و الفلفل الحلو و الفلفل الحار على التوالى .

وفى هذه التجربة كانت فترات الأمان الموصى بها لمبيدى الفنتروثيون و البيريموفوس- ميثيل هى ٣ ، ٢ يوم و ٧ ، ٤ يوم و ٧,٨ ، ٧ يوم بالنسبة لثمار الخيار و الفلفل الحلو والفلفل الحار على التوالى . المتبقى من المبيدين بعد ٨ أيام من المعاملة على ثمار الخيار و الفلفل الحلو و الفلفل الحار كان أقل من الحد المسموح به من قبل لجنة دستور الأغذية و الزراعة (الكودكس). لوحظ أيضاً أن فترات الأمان لمبيد الفنتروثيون بالنسبة لثمار هذه الخضراوات كانت أطول نسبياً من القيم المقابلة لمبيد البيريموفوس- ميثيل.

أوضحت الدراسة أن عملية الغسيل بماء الصنبور بعد يوم واحد من المعاملة أزالت ١٥,٣٨ % ، ٨٧,٢٤% ، ٦٥,٦٥% و ٢٥,٤٥% ، ٧٧,٤٠% ، ٣١,١٣% من متبقيات الفنتروثيون و البيريموفوس- ميثيل من على ثمار الخيار و الفلفل الحلو و الفلفل الحار على التوالى . وكانت النسب المئوية للأ زالة لكلا المبيدين من على ثمار الفلفل الحلو أعلى منها فى حالة ثمار الخيار و الفلفل الحار . ايضاً عملية الغسيل لهذه الثمار لم تسبب أختزال لمتبقيات كلا المبيدين إلى كميات أقل من الحد المسموح به.