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FENITROTHION INSECTICIDE FATE ON AND IN SOME VEGETABLE CROPS

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Abstract

This investigation was carried out to study the fate of fenitrothion insecticide residues on moloukhia, green bean pods and cucumber vegetable crops. Fenitrothion is widely used in Egypt to control economic pests on vegetables. The plan of study included the following: persistence, half life and safe period for consumed moloukhia leaves, green beans pods and cucumber fruits. Also, the effect of washing, blanching, peeling, salting, and drying processes in removing the insecticide residues from the above mentioned vegetable crops.

The results obtained indicated that fenitrothion residues after one hour from application were 107.1649, 18.0787, and 4.4088 ppm on moloukhia leaves, green bean pods and cucumber fruits, respectively. These amounts decreased to 22.3142, 9.5642 and 0.5749 ppm following one day from application, respectivley. After 155 days from treatment the residues became 0.0148, 0.0657 and 0.0057 ppm, respectivley. The half lives values were 10.6, 26.01 and 8.2 hours on moloukhia leaves, green beans pods and cucumber fruits, respectively, while the safe periods for consumption were 5.5, 13.6 2.9 days, respectively.

Effect of different processes in removing fenitrothion residues from moloukhia leaves, green beans pods and cucumber fruits indicated that washing process with tap water removed 85.25% fenitrothion residues on moloukhia leaves, 72.25% on cucumber fruits and 37.44% on green beans pods, respectively. Blanching process removed 80.07% from fenitrothion residues on and in moloukhia leaves and 58.54% on green bean pods. Peeling process of cucumber fruits removed 86.22% from fenitrothion, while after 7 days from salting process of cucumber fruits removed 56.97% and drying process of moloukhia leaves removed 74.13% from fenitrothion residues.

INTRODUCTION

Moloukhia, *Chorchorus olitorius*, Cucumber, *Cucumis sativus* and Green bean, *Phaselous vulgaris*, three popular vegetable crops were the matter of this study. In

Egypt such crops are treated by pesticides in order to control pest infestation. Residues after application of pesticides on vegetable crops should be followed and the waiting periods between application and harvesting should also be recommeneded to be sure that the residues are below tolerance levels before marketing.

Degradation studies of the pesticide residues and processing factors which are expected to affect their presence in and on vegetables were considered. The organophosphorus insecticide, namely fenitrothion is widely used in Egypt to control economic pests especially on vegetables.

This work aimed to study the behaviour of this insecticide residues on and in cucumber, green beans and moloukhia vegetables. The efficiency of washing, salting, peeling, blanching and drying processes in removing the studied insecticide residues were evaluated.

MATERIALS AND METHODS

Pesticide used

Fenitrothion: O-O-dimethyl-0-4-nitro-m-tolyl phosphorodithioate is an organophosphorous insecticide known commerically as Sumithion, Folithion and Accothion. It is used against a wide range of pests i.e. chewing and sucking insect pests. The formulation of Sumithion 50% E.C. produced by Sumitomo chemical, Japan, was used at the rate of one litre in 400 litre of water per faddan (i.e. 500 g a.i.).

Field experiment and sampling

Moloukhia, cucumber and green bean seeds were planted on May 10th, 15th and 23rd 1994, respectively under the normal field conditions and agricultural practices at Kafr El-Sheikh Governorate.

Three plots for each crop were planted in areas of 50 m2, 75m2 and 100 m2 for moloukhia, green beans and cucumber, respectively. These plants were treated by the recommended rates of application of the Ministry of Agriculture, 1993. The plots for each crop were treated with fenitrothion with the recommeded rates. A fourth plot for each crop was left untreated as control. The insecticidal formulation was diluted with water and applied using a knap-sac sprayer equipped with one nozzle as follows:

Moloukhia plants were treated on June 28th 1994; 49 days after planting and

the formulation was diluted at the rate of 400 liters water per feddan. Cucumber plants were treated on July 5th 1994; 51 days after planting and the formulation was diluted by 400 liters water per feddan. Green bean plants were treated on August 6th 1994; 75 days after planting and the formulation was diluted at the rate of 400 liters water per faddan.

Three replicate samples of 300 g each were collected from each crop at intervals of one hour after application (zero time), 1, 3, 6, 9, 12 and 15 days. Clean polyethylene bags were used for preservation of the collected samples. The samples were stored at-20^{o}C in a deep freezer until analysis.

Intentional removal of insecticide residues

Several home and industrial processing which are usually used were evaluated for their efficiencies in removing fenitrothion residues from cuumber fruits, moloukhia leaves and green bean pods. After application of the insecticide with the recommended dose three replicate samples of 300 g each for process were collected after 24 hours and prepared as follow:

Washing with tap water: Cucumber fruits, moloukhia leaves and green bean pods samples were rinsed for three minutes with running tap water, then drained on a clean paper for one hour until dry, and kept in polyethylene bags in a deep freezer until analysis.

Salting of cucumber fruits: Cucumber fruits were cut into samll pieces and packed into half litre glass jars contained 100 ml of 10% NaC1 salt solution and 100 ml of 3% acetic acid solution. The glass jars were kept under room conditions for 7 days before analysis. The samples were kept under deep freezing conditions until analysis.

Peeling of cucumber fruits: Cucumber fruits were peeled manually and the peeled samples were kept in polyethylene bags under deep freezing until analysis.

Blanching of green bean pods and moloukhia leaves: The green bean pods and the leaves of moloukia were cut into small pieces. Then they were placed in a jar filled with boiling water for 2-3 min., then drained and left until they reached room temperature. Then were kept in polyethylene bage under deep freezing until analysis.

Drying of moloukhia leaves: Moloukhia leaves were spread on a clean paper

under room condition for 6-7 days until complete drying and then ground and kept in polyethylene bags under deep freezing conditions until analysis.

Pesticide residues analysis techniques

Extraction

a. Cucumber fruits: Methanol was found to be the best solvent for extracting the insecticide from cucumber fruits. Freezed samples were left until they reached room temperature and then macerated using Waring blender. Fifty grams of the macerated sample were placed in the blender and constant amount of methanol (2 ml/gram plant material) was added to the blender and mixed for 3 min., then filtered through a dry pad of cotton into a 100 ml graduated cylinder to reach half volume. Extracts were shaken in separatory funnel successively three times with 50 ml chloroform each and 40 ml of sodium chloride solution (20%) and then the water phase was discarded. The combined chloroform phases were dried by filteration through a pad of cotton and anhydrous sodium sulphate, then evaporated just to dryness using a rotary evaporator at 40°C.

b. Moloukhia leaves and green bean pods: The freezed samples were left to reach room temperature, then cut into small pieces by using a pairs of scissors and forceps. Fifty grams of the sample were placed into the blender cup with 50 g anhydrous sodium sulphate and 150 ml ethyl acetate, then blended for 3 min. The liquid was decanted through a funnel with a plug of cotton in a graduated cylinder, then evaporated just to dryness by using a rotary evaporator at 40°C.

Cleaning up: It was found that chromatographing the extracts through deactivated florisil (with 6% by weight of water) using benzene for elution is quite efficient and gave good recoveries (Mollhof, 1975).

A 25 mm (i.d.) glass column was prepared by adding successively, a plug of glass wool and 8 g of deactivated florisil (60-100 mesh) and combact throughly. The column was prewashed using 30 ml benzene and drained the level of the solvent down to the top of the florisil. Residue extracts were dissolved in 10 ml of benzene and added to the column. The flask was rinsed five times with 5 ml portions of benzene and added each washing to the column just before the preceding fraction has completely entered the packing. Then, the residues were eluted with 200 ml benzene.

The cleaned up extract was collected into a 500 ml flask, and the solvent was

evaporated just to dryness using a rotary evaporator at 40°C. The residues were quantitatively transferred to standard glass stoppered test tube with ethyl acetate, and the solvent was evaporated just to dryness and the residues were redissolved in the proper volume of ethyl acetate for chromatographic determination.

Gas liquid chromtography determination: A pye Unicam 4500 gas chromatograph equipped with flame photometric detector operated in the phosphorus mode (526 nm filter) was used for determination of fenitrothion insecticide. A pyrex glass column (1.5 m x 4 mm i.d) was packed with 4% S E - 30 + 6% O V-210 on gas chromosorb Q (80-100 mesh).

Temperature degrees were as follow:

Temperature column 230°C.

Detector 240°C.

Injector 235°C.

Gases flow rates were 30 ml/min. for nitrogen, hydrogen and air. Retention time for fenitrothion under these conditions was 5.77 min.

Rate of recoveries of the insecticide on moloukhia leaves, green beans pods, and cucumber fruits were determined by adding known amount of the insecticide to portion of untreated samples and processed as mentioned before. Untreated samples were used as control blank.

Following these techniques, the average rates of recovery for fenitrothion were 80.0, 90.10 and 100% in moloukhia leaves, green bean pods and cucumber fruits, respectively. The results obtained were corrected according to their recovery percentages.

RESULTS AND DISCUSSION

Persistence of fenitrothion insecticide residues on and in vegetable crops

Data in table 1 indicate the amount of fenitrothion residues in moloukhia leaves, green bean pods and cucumber fruits at different intervals after application. The initial deposits of fenitrothion were107. 1649, 18.0787 and 4.4088 ppm on and in moloukhia leaves, green bean pods and cucumber fruits, respectively. These residues decreased after 24 hours to 22.3142, 9.5642 and 0.5749 ppm, respectively.

The residues of this insecticide dropped to 1.8103, 0.2793, 0.1626, 0.0348 and 0.0148 ppm after 3, 6, 9, 12 and 15 days, respectively from treatment with fenitrothion on moloukhia. The corresponding values on green bean pods and cucumber fruits were 5.1262, 1.035, 0.2815, 0.191, 0.0657 ppm and 0.0442, 0.0329, 0.0178, 0.0067, 0.0057 ppm, respectively at the same mentioned intervals. Also, the data indicate that the residues loss was found to be continued on prolonging the time, where the precent loss rate amounted to 79.18, 98.31, 99.74, 99.97 and 99.99% after 1, 3, 6, 12, and 15 days, respectively for moloukhia leaves. The corresponding precent residues loss were 47.10, 71.65, 94.27, 98.44, 99.18, 99.64% and 86.96, 98.99, 99.25, 99.60, 99.85, 99.87% for green bean pods and cucumber fruits, respectively at the same intervals.

The calculated half life values of this insecticide were 10.6, 26.1 and 8.2 hours on moloukhia leaves, green bean pods and cucumber fruits, respectively. While the first day after application showed to be critced in the residue degradation rates.

The level of pesticides residue is affected by many factors i.e. applied dosage, meteorological and biological factors depend on the kind and properties of the plant surface. The obtained results coincide with those reported by Hegazy et al. (1988 b) and Hegazy et al. (1997 a, b and c).

According to (Codex Alimentarius commission, 1990) the maximum residues limites for fenitrothion on the leafy crop spinach (used for moloukia), green bean pods, and cucumber fruits were 0.5, 0.1 and 0.05 ppm, respectively, the corresponding recommended preharvest intervals (PHI) were 5.5, 13.6 and 2.9 days after fenitrothion application for moloukia, green beans and cucumber, respectively.

According to Bates (1979), data on pesticide residues in treated crops are required for the premarket registration of pesticides and for setting maximum residues limits (toxicologically acceptable level) to protect the consumer against the possible health hazards of exposure to pesticides.

Generally, it was found that organophosphorus pesticides for short periods in plant and other environmental constituents (Al-Samariee et al. 1988). The safe period for harvesting the organophosphorus insecticides treated vegetables 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).

Table 1. Residues of fenitrothion on and in vegetables.

Time after application (Days)	Moloukhia leaves		Green bean pods		Cucumber fruits	
	Residues		Residues		Residues	
	ppm	% loss	ppm	% loss	ppm	% loss
Zero time*	107.1649	00.00	18.0787	00.00	4.4088	00.00
1	22.3142	79.18	9.5642	47.10	0.5749	86.96
3	1.8103	98.31	5.1262	71.65	0.0442	98.99
6	0.2793	99.74	1.0354	94.27	0.0329	99.25
9	0.1626	99.85	0.2815	98.44	0.0178	99.60
12	0.0348	99.97	0.1491	99.18	0.0067	99.85
15	0.0148	99.99	0.0657	99.64	0.0057	99.87
RL50 in hours	10.6		26.1		8.2	

* One hour after application

Removal of fenitrothion residues from treated vegetables by some processes

The increasing use of chemical insecticides to control the economic pests which attack field crops has led to increase the pollution of the crops with their toxic residues. Therefore, the need for efficient treatments to reduce or remove such residues became invaluable.

People in Egypt usually use water to wash vegetables before consumption. Other processes also used for vegetables preparation before eating. The efficiency of these processes: washing with tap water, blanching, peeling, drying, and salting were evaluated in removing fenitrothion from vegetables after one day from treatment with this insecticide. However, in Egypt, there are limited studies on the effect of food preparation on the removal of pesticide residues from various crops.

The results in table 2 show the residue levels in ppm and the percent removal of fenitrothion after differnt processing treatments. The residues of fenitrothion on unprocessed moloukhia leaves one day after spraying with the insecticide were 22.3142 ppm. The washing process reduced the residues to 3.28 ppm representing removal of 85.29% of the insecticide residues on moloukhia leaves. The blanching process reduced the residues of fenitrothion to 4.4472 ppm on the same crop. The dried leaves contained 23.0909 ppm of fenitrothion (based on dry weight) which means 74.13% residue removal considering the humidity percentage in moloukhia leaves 75%.

As shown in table 2, the residues of fenitrothion on unprocessed cucumber fruits after one day from spraying with this insecticide was 0.5749 ppm. This value decreased to 0.1597 ppm after washig of cucumber fruits, to 0.0792 ppm after peeling process and to 0.2484 ppm when salting process occurred, and the precents removal were 72.22, 86.22 and 56.79%, respectively. Also, in table 2, the residues of fenitrothion on unprocessed green bean pods after one day from spraying was 9.5642 ppm. The washing and blanching processes reduced the residues to 5.9836, and 3.9656 ppm and the percents removal were 37.44 and 58.54%, respectively.

Table 2. Effect of some different processes on fenitrothion insecticide residues in some vegetable crops.

Crop	Process*	Fenitrothion	
		PPM	% removal
Moloukhia leaves	Unprocessed*	22.3142	00.00
	Washing	3.2818	85.29
	Blanching	4.4472	80.07
	Drying**	23.0909	74.13***
Cucumber fruits	Unprocessed*	0.5749	00.00
	Washing	0.1597	72.22
	peeling	0.0792	86.22
	Salting	0.2484	56.79
Green bean pods	Unprocessed*	9.5642	00.00
	Washing	5.9836	37.44
	Blanching	3.9656	58.54

* After one day from application.

** Calculated on dry weight.

*** The % removal calculation was based on the moisture content in moloukia leaves was 75%.

Heavy use of pesticides on the field crops has begun to receive much attention because residues in food commodities may be hazardous to human health. The present data indicated that the different processing that used in this study had dif-

ferent effects on reducing or removal pesticides residues origenally located on or in the mature fresh leaves or fruits of different cultivars. This variation in their effect depended on the type of pesticides used, type of processing applied and location of residues on the parts applied. Therefore, many researchers have studied how to remove pesticides from food products in the world, Liska and Stadelman (1985), Geisman (1975), Celino and Magallona (1985) and Bessar et al. (1991). The present results also confirm and agree with those obtained and reported by National Canners Association (1967) that peeling fruits removed considerable amounts of pesticides residues not removed by washing treatment. Karogeorgiev (1979) also found that peeling of fruit results in complete disappearance of pesticides residues, within the limit of the methods used in analysis. Washing processes were found to be efficient in removing organophosphorus insecticides from vegtables, Fahey et al. (1969) and (1971), Tantawy et al. (1979), Kamil (1987), Hegazy et al. (1988-a), Bessar et al. (1991) and Ramadan et al. (1992).

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مصير مبيد الفنتروثيون على بعض محاصيل الخضر

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إجرى هذا البحث لدراسة مدى تلوث بعض نباتات الخضر مثل الملوخية والفاصوليا والخيار بمتبقيات الفنتروثيون وهى من المبيدات الفوسفورية التى تستخدم لمكافحة الافات التى تصيب تلك المحاصيل.

وتتضمن خطة الدراسة النقاط التالية.

١. تحليل متبقيات هذا المبيد على اوراق الملوخية وقرون الفاصوليا وثمار الخيار على فترات زمنية مختلفة وحساب قيم زمن نصف العمر للمتبقيات وتحديد فترة الامان لكل مبيد على كل محصول.

٢. دراسه تأثير عمليات الاعداد والتجهيز المختلفة من غسيل وسلق وتقشير وتجفيف على ازالة متبقيات هذا المبيد من على اوراق الملوخية وقرون الفاصوليا وثمار الخيار.

ويمكن تلخيص النتائج المتحصل عليها فيما يلى:

١. تحليل متبقيات الفنتروثيون على اوراق الملوخية وقرون الفاصوليا وثمار الخيار.

اوضحت النتائج ان الكميات المتبقية من مبيد الفنتروثيون بعد ساعة من الرش بالمعدلات الموصى بها كانت ١٦٤٩ ,١٠٧و ١٨,٠٧٨٧ و ٤,٤٠٨٨ جزء فى المليون على اوراق الملوخية وقرون الفاصوليا وثمار الخيار على التوالى. ثم تناقصت هذه الكميات من المتبقى بعد يوم من الرش الى ٢٢,٣١٤٢ و ٩,٥٦٤٢,٠٥٧٤٩ جزء فى المليون على التوالى. ثم أخذت هذه الكميات من المتبقى تقل تدريجيا الى ان وصلت الى ١٤٨,٠٠ و ٦٥٧,٠٠ و ٠,٠٠٥٧ جزء فى المليون بعد ١٥ يوم من الرش على التوالى. وكانت فترات نصف العمر لمتبقيات الفنتروثيون على اوراق الملوخية وقرون الفاصوليا وثمار الخيار ١٠,٧ و ٢٦,١ و ٨,٢ ساعة على التوالى. وامكن تحديد فترات الامان لهذا المبيد بعد الرش حيث كانت ٥,٥ و ١٣,٦ و ٢,٩ يوما بالنسبة لمحصول الملوخية والفاصوليا والخيار على التوالى.

٢. دراسة تأثير عمليات الاعداد والتجهيز المختلفة على ازالة متبقيات مبيد الفنتروثيون من على اوراق الملوخية وقرون الفاصوليا وثمار الخيار.

أوضحت الدراسة ان عملية الغسيل بماء الصنبور ازالت ٨٥,٢٩٪ من متبقيات الفنتروثيون من على اوراق الملوخية، بينما أزالت هذه العملية ٧٢,٢٢٪ من متبقيات هذا المبيد من على ثمار الخيار. أيضا ازالت عملية الغسيل بماء الصنبور ٣٧,٤٤٪ من مبقيات مبيد الفنتروثيون من على قرون الفاصوليا، وان عملية السلق ازالت ٨٠,٠٧٪ من متبقيات هذا المبيد من على اوراق الملوخية و ٥٨,٥٤٪ من على قرون الفاصوليا، وان عمليتا التقشير والتخليل ازالتا ٨٦,٢٢٪ و ٥٦,٧٩٪ من على ثمار الخيار على التوالى. كما ان عملية التجفيف لاوراق الملوخية ازالت ٧٤,١٣٪ من هذا المبيد.