Zagazig J. Agric. Res., Vol. 36 No. (4) 2009 825-836

RESIDUES OF SOME PESTICIDES ON AND IN OKRA PLANTS AND SOIL

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Accepted 22 /8/2009

ABSTRACT: Okra plants, Baladi variety, were subjected to one spray with pirimiphos-methyl, malathion and methomyl insecticides using the recommended rates during 2005 growing season. The residues of the three insecticides were determined on and in green fruits, green leaves of okra plants and soil after spraying. The effect of washing with tap water and blenching of green fruits in boiled water on insecticide residues were also estimated. Summarized results show the following:

The amount of initial deposits of the three tested insecticides in and on green leaves were 52.32, 38.50 and 32.50 mg/kg for pirimiphos –methyl, malathion and methomyl, respectively. These amounts decreased gradually till reached 2.61, 0.81 and 0.46 mg/kg after 14 days of spraying representing 95.01, 97.86 and 98.58% loss of the initial deposits, respectively.

The corresponding deposits in unwashed fruits were 2.7, 2.13 and 0.98 mg/kg for pirimiphos-methyl, malathion and methomyl, respectively. These amounts decreased gradually till reached 0.08, 0.04 and 0.02 mg/kg after 14 days of spraying representing 97. 03, 98.12, and 97.95% loss of initial deposits, respectively. Washing the green okra fruits with tap water decreased the amounts of initial deposits of tested insecticides by 41.11, 29.58 and 27.55% for the three insecticides, respectively. Blenching process reduced the amounts of initial deposited of these insecticides by 84.81, 81.69 and 83.09 %, respectively. The amounts of initial deposits of the tested insecticides in soil were 1.50, 0.98 and 0.48 mg/kg, respectively. Residues decreased gradually till reached 0.09, 0.02 and 0.02mg/kg

after 14 days of spraying showing 94.0, 97.96 and 95.83% loss, respectively. Cooking okra fruits, washed and unwashed fruits, could be however, used for human consumption directly after spray, 10 and 10 days of spray with pirimipho-methyl; 7, 14, and 14 days of spray with malathion and directly after spray with methomyl, insecticide, respectively.

Key words: Pirimiphos- methyl, malathion, methomyl, okra, residues.

INTRODUCTION

Okra is a very important crop, as a human food, in all countries. Okra plants liable to be attacked with different insect pests. The control of these pests is considered an integral part of any strategy. Insecticides are, however, still used in a large scale throughout the world, especially in the developing countries, as a major mean for pest management. Their uses open, however, the possibility of global pollution. Vegetables, like other economic crops, may be exposed to insecticides in various ways, which undoubtedly would contain various levels of insecticide residues. (Ramadan, 1982; Aioub, 1989, 1997 and 2005; Godfred and Osei, 2008 and Zorka and Maja, 2009). It is of great importance to minimize such levels of insecticide residues remaining in crops to the permissible levels. To achieve this, waiting periods between application and harvesting should be established to be sure that, at time of harvesting, the residues are below such tolerance levels.

Such insecticides my be absorbed into soil particles or leached to ground water depending on its physiological properties i.e. the partition coefficient of these toxicants and their metabolites.

Sometimes, initial deposits of insecticides may be removed by washing although the presence of adjuvants counteract removing such deposits (El- Nabrawy and Shalaby, 1992 and Aioub, 1997). Cooking process apparently reduce more amounts of insecticide residues present in insecticide-contaminated vegetable fruits than washing with tap water (Shalaby *et. al.,* 1991).

During insecticidal application, the soil is contaminated by these xenobiotics. Such insecticides may be adsorbed into soil particles or leached to ground water depending on the partition coefficient of these toxicants and their metabolites.

Several investigators assayed the residues of pesticides in edible parts of field and vegetable crops to find out the safety period (Balinov and Shalaby. 1986; El-Shemy and Ramadan, 1987; Ramadan, 1988;-Ramadan and Amir, 1988; Ramadan and El-Shemy, 1990; Shalaby, 1992; Aioub, 1997; Gatwary 2003 and Soliman, 2004).

The present study aimed to determine the residues of pirimiphos-methyl, malathion and methomyl insecticides on and in green fruits, green leaves and soil of okra plants as well as the role of washing and blenching processes in reducing insecticide residues in contaminated okra fruits.

MATERIALSAND METHODS

Field Experiments

Okra seeds were sowing at Kafr-Sakr distract, Sharkia governorate, Egypt on April 4, 2005. The experimental area was divided into plots of 1/200 of feddan each arranged in randomized block design with three replicates for each treatment and untreated control. The normal agricultural practices were adopted. The three used insecticides and their rates in gram active ingredients (a.i.) per feddan were:

1. Pirimiphos-methyl (actelic, 50% EC.); 750 g.
2. Malathion 57% EC; 570 g.
3. Methomyl 90% SP; 270 g.

A Knapsack hand sprayer, fitted with one nozzle was used. Each recommended volume of the used insecticides was diluted with 200 L. water per fed. Each insecticide was sprayed once on July 1, 2005. The untreated control plots were left unsprayed.

Determination of Insecticide Residues

Preparation of samples

Representative samples of green fruits and green leaves of okra and soil were taken 1 h., 1, 3, 7, 10, and 14 days after spraying for residues determinations (at this time the` mature fruits may be collected by farmers for marketing and consumption). Three sub samples of fruits (100 g each), the first one was washed with tap water, and the second was left unwashed while the third one was washed with tap water and boiled in tap water for 20 minutes. At the same time, representative samples

of soil (0.5kg) were taken from the upper 15cm surface layer of insecticide-treated okra field.

Extraction

Pirimiphos-methyl, malathion and methomyl residues were extracted from plant and soil samples according to the method of Macnell *et al.* (1975).

Clean-up

Purification by thin layer chromatography was adopted according to Soliman *et al.* (1982). TLC plates 20×20 cm coated with silica gel GF_{254} (Merck) were used for the clean up of extracted samples.

Residue determination

The residues of pirimiphos-methyl and malathion (organophosphrous insecticides) were determined colorimetrically using the methods of Getz and Watts (1964). The method of Meagher *et al.* (1967) was used for methomyl (carbamate insecticide) residues determination. In these methods the absorbance of the colours were read at wave lengths of 520 and 350 nm for organophosphorus and carabamate insecticides, respectively, using spectronic 20 D spectrophotometer.

Results of the three tested insecticides were corrected using their respective recovery rates in green fruits which were 82.50, 87.92 and 92.30 for pirimiphos-methyl, malathion and methomyl, respectively.

In order to study the rate of degradation and half life period of each of the insecticide used, the steps were conducted according to Gomaa and Belal, 1975).

RESULTS AND DISCUSSION

Data summarized in Tables 1, 2 and 3 represent the amounts of pirimiphos –methyl, malathion and methomyl in green fruits (unwashed, washed and blenched) and green leaves of okra plants and soil.

Results show that the initial deposits of tested insecticides in unwashed green fruits were 2.70, 2.13 and 0.98 ppm for pirimiphos-methyl, malathion and methomyl, respectively. These figures decreased gradually till reached 0.08, 0.04 and 0.02 ppm after 14 days of spray, indicating 97.03, 98.12 and 97.95% loss of the initial deposits, respectively.

Washing the green fruits of okra plants with tap water decrease

Table 1. Residues (in ppm) of pirimiphos-methyl (750 gm a.i./ fed.) on and in fruits and leaves of okra plants and soil

Days after application	Fruits						Green leaves		Soil	
	Un washed		Washed		Blenched					
	ppm	% Dissipation	ppm	% Loss by washing	ppm	% Loss by blenching	ppm	% Dissipation	ppm	% Dissipation
Initial	2.7		1.59	41.11	0.41	84.81	52.32	-	1.5	-
1	1.78	43.07	1.21	32.02	0.18	89.88	28.29	44.02	1.12	25.33
3	1.35	50	1.06	21.48	0.16	88.14	23.02	56	0.31	46
7	0.67	75.18	0.6	10.44	0.05	92.53	14.64	72.01	0.52	65.33
10	0.35	37.03	0.03	8.57	0	100	6.3	87	0.22	85.33
14	0.08	97.03	UND	0	0	100	2.61	95.01	0.09	94

Figures are the average of three replicates.

Initial: sample were taken one hour after application.

UND: undetectable residues.

Table 2. Residues (in ppm) of malathion (570 gm a.i./fed.) on and in green leaves of okra plants and soil

Days after application	Fruits						Leaves		Soil	
	Un washed		Washed		Blenched					
	ppm	% dissipation	ppm	%loss by washing	ppm	%loss by blenching	ppm	% dissipation	ppm	% dissipation
Initial	2.13	-	1.5	29.58	0.39	81.69	38.5	38.5	0.98	-
1	1.45	31.92	1.2	20	0.17	88.27	25.7	33.22	0.78	20.41
3	1.1	48.35	1	12.72	0.15	86.36	19.9	48.38	0.68	30.61
7	0.48	77.46	0.4	10.41	0.04	91.66	12.7	66.93	0.29	70.41
10	0.3	85.91	0.3	6.66	0.03	90	4.92	87.22	0.15	84.69
14	0.04	98.12	0	0	0.01	75	0.81	97.86	0.02	97.96

Figures are the average of three replicates.

Initial: sample were taken one hour after application.

the amount of the initial deposits (zero time) of the tested insecticides by 1.11, 0.63 and 0.27ppm which represent 41.11, 29.58 and 27.55 % loss of the initial deposits of pirimphos-methyl, malathion, and methomyl, respectively.

The capacity of washing in removing the residues of the three sprayed insecticides decreased as time lapsed between the onset of spraying till 2 weeks later; the loss% ranged between 41.11-0.0, 29.58-0.0 and 27.55-0.0 for pirimiphos-methyl, methmoyl and malathion, respectively. The effect of washing process was obvious with pirimiphos-methyl (41.11%) compared with malathion (29.58%) and methmoyl (27.55%) this is due to the differences in partition coffficient.

Data show that cooking process did reduce the amount of insecticide residues in green fruits of okra plants. Blenching of okra fruits in boiled water removed magnitude amounts of insecticides residues. The loss percentage by blenching process ranged between 84.81- 100, 75.00-100 and 83.09-100 in pirimiphos-methyl, malathion and methomyl contaminated okra fruits, respectively.

It is obvious that okra leaves contained higher residues than those obtained with fruits; this may be due to differences in roughness and surface area contaminated with insecticides in the same weight. Amount of residues were 52.32, 38.50. and 32.50 ppm just after spraying for pirimiphos- methyl, malathion and methomyl, respectively. Dissipation percentages were much higher, especially in the first two samples. Such differences may be due to differences in the amount of residues.

In contrary with leaves, initial deposits of pesticides in soil were much lower than those with fruits. Pirimiphos-methyl, malathion and methomyl residues in soil were 1.5, 0.98 and 0.48 ppm, respectively. These amounts decreased gradually by time till reached 0.09, 0.02 and 0.02 ppm after 14 days of spraying. Loss Percentage in residues differed from insecticide to another which reflects the role of physic-chemical properties and sorbance capacity of the tested insecticides.

Concerning the residual half lives of the three tested insecticides in okra plants and soil, results in Table 4 show that pirimiphos- methyl has residual

Table 3. Residues (in ppm) of methomyl (270 gm a.i./fed.) on and in green leaves of okra plants and soil

Days after application	Fruits						Leaves		Soil	
	Unwashed		Washed		Blenched					
	ppm	% dissipation	ppm	% loss by washing	ppm	% loss by blenching	ppm	% dissipation	ppm	% dissipation
Initial	0.98	-	0.71	27.55	0.12	83.09	32.5	-	0.48	-
1	0.73	25.51	0.58	20.54	0.06	89.65	19.3	40.61	0.33	31.25
3	0.49	50	0.44	10.2	0.04	90.9	17.2	47.2	0.26	45.83
7	0.25	74.48	0.24	4	0.024	90	8.06	75.2	0.15	68.75
10	0.15	84.69	0.15	0	0.01	93.33	2.76	91.5	0.08	38.33
14	0.02	97.95	UND	0	UND	0	0.46	98.58	0.02	95.83

Figures are the average of three replicates. Initial: sample were taken one hour after application
UND: undetectable residues.

Table 4. Residual half lives (in days) of pirimiphos-methyl, malathion and methomyl on okra plants and soil

Insecticides	Green fruits		Green leaves	Soil
	Unwashed	Washed		
Pirimiphos -methyl	**3.01**	**4.65**	**2.39**	**3.1**
malathion	**3.43**	**4.16**	**2.32**	**3.6**
Methomyl	**3.17**	**4.31**	**2.4**	**3.4**

half lives 3.01, 4.65, 2.39 and 3.1 days in unwashed, washed green fruits, green leaves and soil, respectively. Residual half lives for malathion were 3.43, 4.16, 2,32 and 3.6 days while with methomyl these values were 3,17, 4.31, 2.4 and 3.4 days, respectively.

Maximum residue levels (MRL) of pirimiphos-methyl, malathion and methomyl in some vegetable crops were 0.5, 0.1 and 2 ppm, respectively as published by (CAC/ PR, 1997). Comparing these figures with those presented in Tables 1, 2 and 3 show that cooking okra fruits, washed and unwashed fruits, could be however, used for human consumption directly after spray, 10 and 10 days of spray with pirimiphos- methyl; 7, 14 and 14 days of spray with malathion and directly after spray with methomyl insecticide, respectively.

Similar results were obtained by Aioub (1997 and 1998); Gatwary 2003 and Soliman 2004; Godfred and Osei (2008) studied the contamination and health risk hazards of organophosphrous pesticides residues in vegetables Ethyl- chlorpyrifos, observed at an average of 0.211± 0.010 mg /kg. in 42 % of tomato, 0.096 ± 0.035 mg/ kg in 10% of eggplant and 0.021 ± 0.013 mg/kg in 16% pepper was below the 0.5 mg/ kg MRL. Dichlorvos was the most frequently detected residue in all the samples analyzed. Levels of malathion in tomato (0.12± 0.101mg/kg) and pepper (0.143±0.42mg/kg) exceeded the MRL of 0.1mg/kg. Health risks were found to be associated with methyl-chlorpyrifos, dichlorvos, monocrotophos and omethioate in eggplant. Routine monitoring of these pollutants in food items is required to prevent control and reduce the pollution and to minimize health risks.

Zorka and Maja (2009): studied the persistence of pesticide residues in market foods in Croatia. Pesticide residues were determined by gas chromatography with mass selective detector (GC-MSD). Sample extract was cleaned up using gel permeation chromatography (GPC). In 66.7% of the samples no residues were found, 25.8% of samples contained pesticide residues at or below MRL, and 7.5% of samples contained pesticide residues above MRL. Most frequently found pesticide were imazalil (found in 35 samples) and chlorpyrifos (found in 24 samples).

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متبقيات بعض مبيدات الآفات على وفى نباتات البامية والتربة

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تم رش نباتات البامية رشة واحدة بثلاث مبيدات حشرية (بيريميفوس- ميثيل، ملاثيون وميثوميل) بالمعدلات الموصى باستخدامها خلال موسم النمو ٢٠٠٥ وقد تم تقدير متبقيات هذه المبيدات عقب الرش مباشرة وعلى فترات من الرش على وفى ثمار البامية والأوراق الخضراء وكذلك فى التربة. كما تم دراسة تأثير عمليات الغسيل بماء الصنبور والسلق فى ماء مغلى للثمار الخضراء على متبقيات المبيدات فيها.

أوضحت النتائج المتحصل عليها ما يلى:

كمية المترسب الأولى على وفى الأوراق كانت ٥٢,٣٢ ، ٣٨,٥٠ ، ٣٢,٥٠ملجم/كجم للبيريميفوس- ميثيل وملاثيون وميثوميل على التوالى، تناقصت هذه الكميات تدريجيا حتى وصلت إلى ٢,٦١ ، ٠,٨١ ، ٠,٤٦ملجم / كجم بعد ١٤يوم من الرش مسجلة نسبة فقد قدرها ٩٥,٠١، ٩٧,٨٦و٩٨,٥٨% على التوالى من المترسب الأولى. كمية المترسب الأولى فى الثمار الغير مغسولة كانت ٢,٧، ٢,١٣ و ٠,٩٨ملجم/كجم للمبيدات المختبرة على التوالى، وتناقصت هذه الكميات تدريجيا بمرور الوقت حتى وصلت ٠,٠٨، ٠,٠٤، ٠,٠٢ ملجم / كجم بعد ١٤ يوم من الرش مسجلة نسبة فقد قدرها ٩٧,٠٣، ٩٨,١٢و ١٧,٩٥% من المترسب الأولى على التوالى. غسيل ثمار البامية بماء الصنبور أحدث انخفاضا فى كمية المترسب الأولى بمقدار ٤١,١١،٢٩,٥٨ و٢٧,٥٥% لمبيدات بيريميفوس- ميثيل، ملاثيون وميثوميل على التوالى. عملية السلق للثمار الخضراء أحدثت نقصا فى كمية المترسب الأولى بمقدار ٨٤,٨١، ٨١,٦٩ و ٨٣,٠٩% على التوالى. كمية المترسب الأولى للمبيدات المختبرة فى التربة كانت ١,٥، ٠,٩٨ و٠,٤٨ملجم/كجم على التوالى، تناقصت المتبقيات تدريجيا حتى وصلت إلى ٠,٩، ٠,٠٢، ٠,٠٢ ملجم/كجم بعد ١٤ يوم من الرش مسجلة نسبة فقد مقدارها ٩٤، ٩٧,٩٦و ٩٥,٨٣ % . أوضحت النتائج أن ثمار البامية المسلوقة والمغسولة والغير مغسولة يمكن استخدامها للاستهلاك الآدمى بأمان بعد الرش مباشرة ، ١٠، ١٠ أيام من الرش مع بيريميفوس- ميثيل و بعد٧ ،١٤، ١٤ يوم من الرش مع مبيد ملاثيون ومباشرة بعد الرش مع مبيد ميثوميل على التوالى.