# MONITORING OF PESTICIDE RESIDUES ON GREEN PEPPER, POTATOES, *Vicia faba*, GREEN BEAN, AND GREEN PEAS IN GAZA GOVERNORATES (PNA) PALESTINE

# By

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#### ABSTARCT

Pesticide residues were monitored in samples of harvested green pepper, potato tubers, and legumes (green bean, green peas, and Vicia faba), from six locations in Palestine. The FPD, ECD, and M.S. chromatographic techniques were used to detect, confirm and quantify any of the detectable pesticide residues in these food commodities. Scarcely few pesticide residues were detected using the FPD technique in the analysis of all the studied vegetables, where only chlorpyrifos was detected by FPD in the potatoe tubers, but at levels lower than the Maximum Residue Limits (MRL) value. On the other hand, majority of the pesticide residues were detected by the ECD technique, with higher number of residues on green pepper followed by the potatoe tubers, and the least were on the three legumes. The ECD pesticide residues which exceeded the MRL's in potatoe tubers were triademenol, B-endosulfan in both Khan Younis and Gaza locations. Similarly, the following ECD pesticide residues on green pepper and Vicia faba were exceeding the MRL values:- hexacollazole, B-endasuslan, and triadimenol The rest of

the detected residues were far helow the MRL values and thus do not cause any health hazard.

#### INTRODUCTION

Cai et al., (1995), described a rapid and efficient multiresidue extraction procedure using ethyl acetate and sodium sulfate which was applied to the analysis of organophosphate insecticides (OP's). The limit of quantification of the analytical method was estimated as 0-01 mg/kg (p.p.m) for diazinon, methamidophos and malathion, 0.03 mg/kg (ppm) for chlorpyrifos, parathion, parathion-methyl and dimethoate; and 0.1 ppm for monocrotophos. Experiments showed that potentially, it should be possible to develop a rapid and universally applicable method for OP residues in different matrices.(Anonymous, 1994).

Ahmed, and Ismail (1995) reported an accurate and rapid HPLC method which was developed to monitor residues of methomyl on strawberries, tomatoes, and cucumber, Residues reached levels of 0.55; 0.2; and 0.6 mg/kg at 7 days after application on strawberries, tomatoes; and cucumber. The results showed that rinsing treated fruits with tap water, rewoved considerable amounts of methomyl which is water soluble.

Leone et al., (1995) studied the decay of primicarb, chlorpyrifosmethyl, and penconazole pesticides which were widely used in Puglia, Italy; for the protection of artichokes, using GLC with capillary column and a nitrogen/ phosphorous detector. The residue at the end of the prebarvest interval were below those permitted by the Italian law.

Mukherjee and Gopal (1996) monitored the insecticide residues in baby food, animal feed; and vegetables by gas liquid chromatography. Apart from the levels of DDT in baby milk (> 0.05 ug/gm in 5 of 6 brands); the levels of the other compounds were negligible.

Singh et al., (1995) carried a study on the dissipation of malathion in chillies for determination of safety preharvest periods. Field

experiments were carried out in India during the kharif of 1991/1992. The crop was sprayed with malathion at 625 and 1250 gai/h at the fruiting stage. Samples were collected at 0, 1, 3, and 5 days and analyzed for malathion residues. The results revealed that a 2-r 3-day waiting period was requind for the safe consumption of capsicum. Decontamination processes such as washing and cooking, removed malathion residues to below the detectable linits even from the day zero samples.

Fermandez Alba *et al.*, (1996) described an HPIC method for the determination of imidaclopid residues in vegetables at levels ranging from 0.01 to 0.60 mg/kg.

Lee et al., (1996) determined the methamidophos residues in food remnants. He reported that over the past 7 years, acute food poisoning cases arising from the consumption of methamidophos-tainted vegetables had occurred sporadically in Hong Kong. To enable prompt remedial and regulatory actions to be taken, a simple and rapid gas chromatographic method was developed to determine methamidophos in food remnants. The method was also applicable to the determination of aceplrate, dimethoate, omethoate, and trichlorfon. Method detection limits ranged from 0.02 to 0.04 mg/kg. In 1994, 75 food poisoning cases in Hong Kong were suspected to be related to the consumption of methamidophos-tainted vegetables. Thirteen food remnants were received for analysis, four of them were found to contain high concentrations of methamidephos.

A flow injection analysis system using immobilized acetylcholinesterase was used to detect methamidophos in vegetable extracts. Acetyl cholinesterase was immobilized onto magnetic particles, which could easily be separated from the test sample. Methamidophas was detected in lettuce and cabbage at 12 and 3 mg/kg vegetable material respectively (Lui et al., 1997).

The persistence of wettable powder formulation of endosulfan and chlorpyifos, and an emulsifiable concentrate of chlorpyrifos, was studied on apples following two sprays. Residues were analysed by gas chronratography using electron capture detection while endosulfan persisted for 10 days after the last spray application, chlorpyrifos was less persistent. (Nath et al., 1997).

Two methods for eliciting pesticide residues in fruits, vegetables and grains are described with the intention to compare the ability of laboratories within the European Union to carry them out effectively (Reynolds *et al.*, 1997).

Criteria for evaluating laboratories for their involvement in the Italian monitoring network of the Ministry of Agriculture on Pesticide residues in food was emphasized (Leandri et al., 1997).

The detection limit for phenothrin by ECD-GC and ion-trap GC/MS by this method was 0.01 ppm; While the detection limit for phenothrin metabolite 3-phenoxybenzoic acid PBA by ECD-GC was 0.001 ppm. An actual surveillance analysis of 6 agricultural products imported from October to December 1994 indicated no phenothrin but PBA was detected in all agricultural products analyzed (Hirahara et al., 1997).

Approximately 200 citrus samples from markets of the Valencian Community (Spain) were analyzed to establish their residue levels of 12 OP's residues during the 1994/1995 campaign. A total of 32-25% of the samples contained pesticide residues and 6.9% exceeded the European Union Maximum Residue Levels (MRL's). The pesticides found in the samples with residues above MRL's were carbophenothrin, ethion, methidathion, and methyl parathion; (Torres et al., 1997). Recently Safi et al., (2000) monitored the pesticide residues on cucumber, strawberries and tomatoes in Palestine. They used the GC-ECD, GC-FPD, and GC-MC- techniques to detect and confirm the pesticide residues on these vegetable crops. Generally, tomatoes showed the least number and levels of residues, while strawberries showed greater number and levels of residues especially by GC-MS technique. Penconazole was the more abundant residue on both cucumber and strawberries followed by tridimenol.

The present study is devoted to monitor the pesticide residues on green pepper, potatoes, *Vicia foba*, green bean, and green peas in different areas in Gaza, Governorate (PNA) using GC-CD, GC-FPD, anal GC-MS techniques for detection, confirmation and quantification of the pesticide residues.

### **MATERIAL AND METHODS**

### Samples:

Twenty one fruit Samples of green pepper, potatoes, Vichia faba, green beans and green peas, were collected from the harvest during the season 1998/1999 at the following six different localities in Gaza Governortes (PNA)

1- Beat Hanon 2- Jabalia

3- Gaza

4-El-Nusirat 5- Deer El-Balah

6- Khan Younis

The samples were kept in paper bags in a refrigerator until processed for the multiresidue analysis of pesticides..

# Sample Processing and Extraction:

Fifteen gm. Samples of each fruit type were weighed and transferred to a homogenizer where it was mixed with 30 ml. acetone, and then 30 ml of petroleum ether + 30 ml methylene chloride, and the mixture was homogenized for one minute. Then the homogenate was centrifuged at 4000 r.p.:n for 10 minutes at 22°C. Aliquots each of 25 ml, of the supernatant were subjected to evaporation then was mixed with 1 ml of the mixture of (90% isooctane + acetone 10%) then the solution was well mixed in a vortex. The resulting solution then became ready for injection in the Gas chromatograph.

# Solvent and Chemical Reagents:

The used solvents and chemical reagents were of gas chromatography grade purity and were provided by Sigma Co.

# Analytical Equipment:

Gas chromatograph with electron capture detector (ECD) and/or flame photometric detector (FPD) mamfactured by Shimadzu Co. was used. The Shimadzu apparatus, equipped with the ECD was used for detection and quantification of the halogen containing pesticides. The FPD detector was used for identification and quantification of the other compounds mainly the organophosphorous compounds.

Confirmation of the detected residues was carried out using the G.C.M.S. Shimadzo. Reference samples were used as standards for both calibration and confirmation purposes. The analytical procedure was applied according to: G.C. Methods, in the US Food and Drug Administration, Pesticide analytical manual, Multiresidue methods 1994; and the methods described by Mills, P.A. et al., (1963). J. Assoc. of (Agric. Chem. 46: 186-191. Reference samples were used as standards for calibration and confirmation purposes. The maximum residue limits (MRL's) were adopted from the Codex AlimIntarius. Commission, Joint FAO/WHO Food Standard Programme (1998) because until now there is no Palestinian Directory of Pesticides.

### RESULTS AND DISCUSSION

#### Green Pepper:

Table(1), present the pesticide residues data detected and quantified on Green pepper crop using GC-FPD, GC-ECD; and GC-MC techniques respectively. The GC-FPD analysis did not reveal the presence of any residues while by the GC-ECD several pesticide residues were monitored (Table 1).

However the detected levels were below the maximum residue limit (MRL) in most cases except for the fungicide Hexaconazole which proved to be present on green pepper in the three locations: Der El-Balah, Jabalia; and Beit Labya. Levels of Hexaconazole residues were actually threatening seriously because their magritude was more than 200 folds of the MRL by the Codex (1998).

Simultaneously, triadimenol was present at 3 fold level on green pepper at Beit Labya. On the other hand the insecticide B-endosulfan residues were quite abundant on green pepper in four locations at levels almost equal or slightly exceeding the MRL in Der El-Balah; Jabalia; Beit Labya; and Beit Hanon.

The GC-MS detection (Table 1), indicated also that the fungicide myclobutanil was the only residue present on green pepper crop in the four locations: Der El-Balah; Jabalia; Beit Labya and Beit Hanoun.

#### Potatoe Tubers:

The monitored pesticide residues in potato tubers are shown in Table 2 using the GC-FPD; GC-ECD; and GC-MS chromatographic techniques.

The GC-FPD analysis revealed only the presence of chlorpyrifos residues on potato tubers in the two locations of Khan Younis, and Gaza-However the levels of chlorpyrifos are far beyond the MRL values of the compound according to the WHO MRL values (WHO, 1997), and Codex (1998).

On the contrary the GC-ECD analysis proved the presence of residues of the insecticide endosulfan isomers and metabolites, in addition to the fungicides triadimenol, prodione, dichlofluanid, vinclozoline, and bromopropylate. Of the 18 residues detected on potato tubars in the six sampled locations, only four were exceeding the MRL values. Triadimenol in both Khan Younis and Gaza were exceeding the MRL. In the same two sites, B-endosulfan was slightly exceeding the MRL value (Table 2).

The GC-MS analysis detected three residues on potato tuber only at Khan Younis. Chlorpyrifos, triadimenol I, and triadimenol II. were detected, however, the level of chlorpyrifos was about six fold the MRL. It is worthy to notice that chlorpyrifos was also detected on potato tubers using the FPD-technique at both the same site at Khan Younis, and also at Gaza as previously shown (Table 2). Thus the GCMS results confirm the presence of chlorpyrifos residues.

Table (1): GC-FPD, GC-ECD and GC-MS Pesticit

				<del></del>	
Area	Detected	t <sub>R</sub> .	Residue	MIR L's	Detected
	Pesticides	(min.)	<b>ා</b> කිරුවියා	mg/kg	Pesticides
		GC	-FPD		
Khan Younis	עא	<u> </u>	-		Chorothaloni
}	j		İ		Dichlofhuanid
	]	1	1	<b>i</b>	'
Gaza	N.D	-			Chorothalonii
	i i				ce-Endosolfan
		ļ	1	<b>i</b> .	β- Findosplfan
	•		•		
Der El-Balah	N.D	-	-	-	o-Endosulfa
		İ	į.		Hexaconazole
	ļ		1	}	B-Endosulfan
	1		}		Iprodione
	ł		}		Endosulfan-Sulf
•			[		THOO STUDENTS OFF
Jabalia	N.D				cc-Endosulfan
		1			Hexaconazote
i		ĺ	<b>!</b>	1	
	ļ	ļ	ł	1	[1-Endosulfan
ļ		1	<b>[</b>		[prodione
		ł	l		Endosulfar-Sulf
			İ		mercust of dis
Beit Lahya	ND	[ .	i -	] -	Triadimenol
·			İ		ca-Endesnifan
					Henaconazole
					β-Endoselfan
			]	}	Iprodiane
	•			<u> </u>	Endosulfan-Sulf
Beit Harnoun	N.D	_	1		
	40 1100		~	-	α-Endosulfan
					β-Endosulfan
					Iprodione
<b>,</b>		ì			Endosnifan-Sulf
t .		₹		[ -	

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Residues on Green Pepper in Gaza Governorates, PNA.

t <sub>g.</sub> (min.)	Residue ug/gm	MR L's Mg/kg	Detected Pesticides	ty. (min.)	Residue ng/gm	MR L'i
GC-EC		147E-14	1 0000000	GC-M		
11.98	0.009	5.0	N.D		7	1 -
13.40	0.033	2.0	11.2		_	
11.98	0.008	5.0	N.D	_	_	-
15.35	0.005	0.5	ļ		•	
17.0	0.064	0.5	•			
15.35	0.161	0,5	Myclobutanil	12.17		0.5
15.53	0.246	0.01	Velt-montami	1124.11	· -	4
17.0	0.527	0.5				
17.50	0.160	5,0				i
18.70	0.312	0.5				
15.35	0.159	0,5	Myclobutanil	12.17	_	0.5
15,53	0241	0.01	1	1 #4.1 r		1 4
17.0	0.512	0.5	[ ]			ł
17.5	0.150	5.0				
18.70	0.287	0,5				
14.5	0.321	0.1	Myclebutanil	12.17	•-	0.5
15.35	0.141	0.5				
15.53	0.225	0.01		ĺ		
17.0	0.438	0.5				
17.5	0.141	5,0				
18.70	0.116	0.5	[			
15.35	0.156	0.5	Myclobutanil	12.17		0.5
17.00	0.511	0.5		1	j	
17.50	0.159	0.5	,	i		
18.70	0.134	0.5	<b>f</b> [	i		

Table (3): GC-(MS), GC-ECD and GC-MS Pesticite

Arca	Detected Pesticides	t <sub>E</sub> . Residue (min.) ng/gm GC-FPD		MRL's mg/kg	Detected Pesticides
Mid-Zone	N.D	-		-	Hexaconazole
Gaza	N.Đ	-	-	•	N.D
North Zone	N.D	-			N.D

Table (4): GC Pesticide Residues On

Ares	Detected Pesticides	t <sub>Be</sub> (min.) GC-	Residue ug/gm FPD	MRL's mg/kg	Detected Pesticides
Mid-Zone	N.D	-	-	-	Endosulfan-Sulf.
G±28	N.D	-	-	•	lprodione
North Zone	N.D		-	-	Iprodione

Table (5): GC -FPD- GC- ECD and GC-MS Pesticide

Area	Detected Pesticides	t <sub>R</sub> . (mān.)	Residue ug/gm	MR L's mg/kg	Detected Pesticides
		GC-	FPD		
Mid-Zone	N.D	_	•	-	Vinclozolia
Gazz	N.D	-		, ,	Hexacsaszele
North Zone	N.D	- (		-	Hexaconazola

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Residues on Green Pepper in Gaza Governorates, PNA.

_			4			
t <sub>R</sub> . (mid.)	Residue ng/gm	MRL's mg/kg	Detected Pesticides	t <sub>B</sub> , (min.)	Residue ug/gm	MRL's mg/kg
GC-E	CD			Ų,	C-W2	<del></del>
15.53	0.012	0.1	N.D	-		-
-	-		N.D	-	-	
	_	-	N.D	<u> </u>		

# Green Pease In Palestine, Season 1998/1999.

t <sub>k</sub> . (min.)	Residue ng/gm	MR L's mg/kg	Detected Pesticides	t <sub>r.</sub> (min.)	Residue ug/gm	MR L's mg/kg
GC-ECD	Annual Little and product only of the spice.	<u> </u>		GC	-MS	
18.70	0.226	0.5	N.D		<u> </u>	•
17.50	0.086	50	N.D	-	·	-
17.50	0.026	5.0	N.D			

# Residues On Vicia faba In Palestine, Season 1998/1999.

t <sub>R</sub> . (min.) GC-EC	Residae ug/gm D	MRL's mg/kg	Detected Pesticides	t <sub>E</sub> . (min.) GC	Residue ug/gm -MS	MRL's mg/kg
12.60	0.007	0.1	N.D	•		-
15.53	0.075	0.01	N.D		-	-
15.53	0.052	0.01	N.D	-		·

Table (2): GC-FPD, GC-ECD and GC-MS Pesticide Residues on Potato in Gaza Governorates, PNA.

Area	Detected Pesticides	t <sub>R</sub> . (mia.)	Residue ug/gm	MR L's mg/kg	Detected Pesticides
Khan	Chi	GC-F	1	1	
Younis	Chlorpyrifo 3	14.20	0.024	0.05	Triadimenol α-Endosulfan β-Endosulfan Iprodione I Endosulfan-sulf Bromopropylate
Gaza	Chlorpyrifo	14.20	0.0001	0.05	Triadimenol
Der El-	]	-	-	-	cx-Endosulfan
Balah	N.D				β-Endosulfan Iprodione J Endosulfan-sulf Bromopropylate
Jahulia	N.D				Vinclozolin Dichlofiuanid Triadimenol
			į	ļ	Iprodione I
Beit Lahya	N.D			1	Iprodione I
Beit Hamoun	N.D				Iprodione I

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t <sub>R</sub> . (min.)	Residue ug/gm	MR L's mg/kg	Detected Pesticides	t <sub>ឆ</sub> , (ការព.)	Residue ug/gm	MR L's mg/kg
GC-ECD	1	<u> </u>		GC-MS		in Sark
14 50	0.351	0.1	Chlerpyrifos	10 76	0.299	0.05
15.35	0 179	0.5			1 4.277	1 110
170	0.576	0.5				1
17.50	0.055	5.0	•	•		
18.71	0.053	5.5			•	
21.30	0.047	1.0				1
14.50	0.286	0.1	Triadimenol I	11.54	0.170	5.0
15.35	0.139	0.5	Triadimenol II	11.63	0.235	5.0
17.0	0.503	0.5				1
17.50	0.029	5.0				
18.71	0.024	0.5				1
21.30	0.056	1.0		ļ		1
12.69	0.008	0.1	N.D			
13.40	0.066	2.0		Ì		
14.50	0.611	0.1				
17,50	0.008	0.1	N.D	ĺ		
17.50	0.020	0.1	N.D			
17.50	0.005	0 I	N.D		!	

### Green Bean, Green pease, and Vicia fobae:

The pesticide residues on green bean was analyzed by the three techniques of GC-FPD; GC-ECD; and GC-MS as shown in Table 3. The only detected pesticides was bexaconazole by the GC-ECD; in the Midzone (Der El-Balah and El-Nussairat). However, the residue level was below the MRL. All the rest of samples did not show any detected pesticide residues (Tables).

Green pease sumples analytical data are presented in Table (4) for the GC-FPD; GC-ECD; and GC-MS techniques. GC-EPD-did not show any pesticide residues detected, while by the GC-ECD (Table 4), the insecticide endosuflan – sulfate metabolite, and prodione were monitored in all the locations. However, the levels were below the MRL values.

The Vicia faba samples analyzed by the GC-FPD; GC-ECD; and GC-MS techniques are shown in Table 5. The only detected pesticides were vinclozoline in the Mid Zone and hexaconazole in Gaza & North Zone on Vicia faba using the ECD technique. It was also obvious that the residues of bexaconazole were exceeding the MRL values.

Generally, it was clear that the monitored pesticide residues were mostly detected by ECD tecnique because most of the compounds were either chlorinated aromatics or containing active moieties potentially captured by the GC-ECD. Besides, most of the positively detected pesticides were below the MRL's. This finding is similar to the results of Torres et al., (1997) who concluded that only 32.25% of the analyzed citrus fruit samples showed detected residues, and only 6.9% of the samples had exceeded the MRL values of the OP's.

The number of pesticide residues were mostly detected on green pepper fruits, followed by potato tubers, and the least number and levels were detected on green legumes (bean, pease, and *Vicia faba*. However, no OP residues were detected. Similar data were reported by Singh *et al.*, (1995). The two insecticides detected were endosulfan (its isomers and metabolites), and chlorpyrifos. However only B-endosulfan was more abundant and showed more cases of higher levels exceeding the MRL. The result coincides with the finding of Nath *et al.*, (1997) who

concluded that endosulfan persisted for 10 days, while chlorpyrifos was less persistent. Leone et al., (1995) stated that the residue amounts detected at the end of the preharvest interval were below those permitted on vegetables. Mukherjee and Gopal (1996) indicated that the levels of pesticides detected in baby food, animal feed, and vegetables were below the MRL values and were not considered to be a possible health hazard.

However, it should be emphasized that some of the plant systemic compounds of high acute toxicity such as methamidophos represent a real health hazard to the consumers. This was demonustrated by Lee et al., (1996) in Hong Kong where 75 food poisoning cases were recorded due to residues of the methamidophos. The health adverse effect of methamidophos is more intensified by knowing that it is a delayed neuropathic agent which should be banned worldwide.

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# المنخص العربي

رصد متبقيات المبيدات في الفلفل الاخضر - البطاطس - القول - القاصوليا والبسلة الخضراء في قطاع غزة - فلسطين

جمال صافى ' ' ' - نصر ابو فول ' ' ' - ياسر النحال ' - عبد الخالق السباعي "

۱- كلية الزراعة - جامعة الارهر بغزة -- قطاع غزة -- فلسطين
 ۲- معهد بحوث وحملية البيئة - غزة
 ۳- قسم كيمياء وسمية المبيدات - كلية الزراعة - جامعة اسكندرية - الشاطبي

تم رصد متبقيات المبيدات في عينات من الحصاد الطازج لكل من الفلف الاختسر والبطاطس والثمار الطازجة لمحاصيل الفول والفاصوليا والبسلة الخضراء. وقد تم تمثيل مستة مواقع في قطاع غزة لهذه العينات. وقد تم استخدام التحليل الكروماتوجرافي بالغاز بانواعسها مواقع في قطاع غزة لهذه العينات. وقد تم استخدام التحليل الكروماتوجرافي بالغاز بانواعسها مقارنتها بحدود التركيزات القصوى الدولية. وقد اظهر التحليل باستخدام — FPD عددا معددا من المتبقيات حيث لم يتعرف الاعلى متبقيات مبيد الكلوربيريفوس فسى درنات البطاطس ولكن مستواها اقل من حدود التركيز الاقصى المسموح به MRL. أمما غالبيسة المتبقيات فقد ظهرت باستخدام ECD وكان اعلى تلك المتبقيات في العدد والتركيز في درنات البطاطس وعلى ثمار الفافل الاخضر بينما كان اقل تلك المتبقيات على محاصيل البقول الثلاثة (الفول والفاصوليا والبسلة الخضراء). والمبيدات التسى زادت متبقياتهما عن عنما منطقتسي غزة كانست مبيدات Arliadimenol مبيد Triadimenol على كل من الفلفل الاخضر والفول الاخضر والفول الاخضر والفول الاخضر والفول المحدد من المبيدات التبيدات التبيدات التمرف عليها فكانت اقال المتورد القصوى التركيزات الممموح بها.