

DETERMINATION OF SOME CHLORINATED AND  
ORGANOPHOSPHOROUS INSECTICIDE  
RESIDUES IN THE SOIL

BY

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ABSTRACT

Soil samples, especially in the root zone, were collected from various crop fields at different localities in Egypt. Samples were analyzed to determine residue levels of certain chlorinated and organophosphorous insecticides. Results indicated that, approximately, all the samples contained residues of Lindane, Alpha HCH and DDT. Residues of Dieldrin were detected only in 13.9 % of the total analyzed samples while in 30.6 % for endrin and both were in trace amounts.

For organophosphorous insecticide residues, only malathion was found in 22.2 % of the tested soil samples. High level of residues were recorded in two samples (4.03 % & 6.76 ppm). According to the information gathered during this study, the fields were treated with malathion before collection of the two samples. None of the samples contained pirimiphos-methyl residues.

### INTRODUCTION

Pesticides are reactive compounds. The amount of residues that accumulates in soil depends on many factors. Soil and climatic conditions, e.g., clay and organic matter content, moisture and temperature. In addition, the physicochemical properties of the insecticide, e.g., volatility and stability to chemical and microbial degradation, play an important role (Harris, 1972). Insecticides, in general, known to be very soluble in water tended to be less active as soil insecticides (Harris and mazurek; 1966; Harris and Turnbull, 1977).

Sears and Chapman (1979) reported that residue analyses were performed on samples of turfgrass treated with chlordane, diazinon, chlorpyrifos and CGA 12223, when applied under optimum conditions at rates normally recommended for control of soil insects which damage turf. Subsequent, samples were taken at intervals up to 56 days after treatment and analyzed for insecticide residues in the grass-thatch layer, in the root zone, and in the underlying soil. Approximately 60 % of the chlordane was recovered from the grass-thatch layer after 56 days while only 9 % of chlorpyrifos remained at that time. Diazinon and CGA 1223 disappeared within 14 days.

Talekar et al. (1983) investigated the persistence of selected insecticides after seasonal (spring and fall) applications to silt loam soil over a 4 - year period. DDT and dieldrin were applied at 5 Kg / ha; foncphos phorate, and carbofuran were applied at 10 Kg /ha. Insecticide residues were monitored in the top 15 cm of soil during spring and fall before application. Insecticide residues

accumulated after fall applications but degraded rapidly after spring applications. Except for dieldrin, no insecticide accumulated from 1 year to the next. After the first 2 years, total dieldrin residues increased with each application. After 5 years, the recoveries of DDT and dieldrin were 6.6 and 15 % of the amount applied, respectively. Fonofos, phorate and carbofuran residues represented less than 0.2 % of the compounds applied after 5 years.

Insecticide degradation was faster during the hot and rainy months than during cool and dry months. Pesticide residues in soil samples collected from 213 villages in 8 centers in Egypt, showed that dieldrin, DDT, endrin and Lindane were detected in all tested samples. About 23 compounds were detected as breakdown or unknown products. Traces of some organophosphorous compounds were also detected in more than 50 % of the samples (Abdel-Gawaad, 1985).

In this study we try to throw some light on the residue levels of certain chlorinated and organophosphorous insecticide residues in the soils at the present time.

### MATERIALS AND METHODS

#### Soil sampling :

Representative soil samples, especially in the root zone (15 - 25 cm depth) were collected from various crop fields at different localities, during the period from September, 1992 to May, 1993. Samples were air dried and sieved through a 2 mm sieve.

#### Extraction procedure :

Samples (25 g soil) were extracted with acetone (100 ml) for an hour in a reciprocal shaker. The mixture was filtered through a funnel fitted with sharkskin paper and two

spoons  $\text{Na}_2\text{SO}_4$ . Fifty ml of the filtrate was transferred into 250 ml round-bottom flask containing 0.5 ml n-hexadecane and the volume was reduced to about 1 ml with a rotary evaporator.

**Clean up procedure :**

Four grams celite was added into the same 250 ml round-bottom flask, mixed well, and all the content was transferred into chromatographic column (equipped with a plug of purified cotton and 1 cm layer of celite). The column was eluted with 25 ml propylene carbonate and the eluate was collected in a 250 ml separatory funnel containing 10 ml petroleum ether, 175 ml  $\text{Na}_2\text{SO}_4$  2 %, water solution was added to the eluate and the separatory funnel was shaken for 2 minutes.

The upper organic layer was eluted in a column containing a plug of purified cotton, a mixture of 9.3 g  $\text{Al}_2\text{O}_3$  + 0.7 g Fuller's earth and 1 cm layer of anhydrous sodium sulfate in a glass filter on the top of the column.

The chromatographic column was eluted by 40 ml of petroleum ether 40 - 60 °C for the chlorinated pesticides. The eluate was collected in a 250 ml round-bottom flask contains 0.5 ml n-hexadecane and was evaporated to dryness with a rotary evaporator. The residues were transferred with n-hexane in 10 ml volumetric flask and the volume was adjusted for injection.

For organophosphorous residues, the same column was eluted by 40 ml mixture of dichloromethane : acetone : benzol in a ratio 5 : 1 : 1. The eluate was collected in the same flask (containing 0.5 ml n-hexadecane) and was evaporated to dryness. Organophosphorous residues were transferred with n-hexane in 10 ml volumetric flask to adjust the volume. All solvents and chemicals are analytical grades.

**Gas-liquied chromatographic (GLC) analysis :**

Hewlett packard series II gas chromatograph equipped with electron capture detector and nitrogen phosphorous detector, and provided with Hewlett packard HP 3396. Integrator was used for determination of organochlorine and organophosphorous insecticide residues. The parameters were as follows : silanized pyrex glass column (1.8 m X 2 mm ID) packed with 1.5 % OV - 17 + 1,95 % OV 202 on chromosorb WHP, 80 / 100 mesh.

For organchlorine analysis, column temperature, was 195 °C, detector temperature was 300 °C, injector temperature was 230 °C, nitrogen (carrier gas ) was 25 ml / minutes.

For organophosphate analysis, column temperature 195 °C, detector temperature 220 °C, injector temperature 230 °C, nitrogen (carrier gas) 25 ml / min., hydrogen 3 - 4 ml / min., air 100 ml / min.

Samples injection were aleternated with injections of standards. Preliminary results from recovery studies, yielded approximately 85 -90 % of chlorinated pesticides (Lindane, Alpha HCH, total DDT, Endrin and Dieldrin) and 84 - 86 % of organophosphorous pesticides (malathion and pirimiphos - methyl).

**RESULTS AND DISCUSSION**

Results of some chlorinated and organophosphorous insecticide residues in the soil of root zone from various crop fields are recorded in Tables (1).

For chlorinated insecticide residues, results show that approximately all soil samples contained residues of Lindane, Alpha HCH and total DDT. Residue levels of Lindane were in the range of 0.001 - 0.05 ppm in all

analyzed samples. Residue level were 0.001-0.32 ppm for Alpha HCH, while they were in the range of 0.001 - 0.45 ppm for total DDT.

Dieldrin residues were found in five samples, representing 13.9% of the total samples analyzed. Residue level were in trace amounts ranged between 0.001 - 0.002 ppm.

Endrin residues were detected in 11 samples, representing 30.6% of the total samples analyzed, in also trace levels ranged between 0.001 - 0.01 ppm.

Malathion was detected in eight samples, representing 22.2 % of the total samples analyzed in the levels of 0.02 - 6.76 ppm. The soil samples contained 4.03 and 6.76 ppm, were treated with malathion to control cabbage pests, according to the gathered information.

Results obtained from this study showed that pesticide residue levels in the soil were much lower than those obtained by Abdel-Gawaad (1985). This may be due to the decreasing use of the chlorinated pesticides in the last five years.

Pesticide residue levels varied from soil to another in the same location, and that could be attributed to soil and climatic conditions, the physio-chemical properties of the insecticides (Harris, 1972).

#### ACKNOWLEDGEMENT

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Table (1): Chlorinated and organophosphorus pesticide residue levels in soil. Samples collected during (1992-1993).

Localities of samples	Field	Chlorinated residues (ppm)					Organophosphorus residues (ppm)	
		Lindane	Alpha HCH	Total DDT	Dield- rin	Endrin	Malathion	Pyrimiphos- methyl
Kafer-El-Dawer, Behera	Cabbage	0.002	0.004	0.020	-	-	0.090	-
	Maize	0.010	0.002	0.030	-	0.002	-	-
	Clover	0.001	0.020	0.005	-	-	-	-
	Bean	0.001	-	0.015	-	-	0.038	-
Abo-Homrus, Behara	Wheat	0.001	0.005	0.052	-	-	0.076	-
	Vegetable	0.003	0.010	0.002	0.002	-	-	-
Shebin-El-Koum Menofia	Orange	0.010	0.010	-	-	-	-	-
	Vegetable	0.004	0.004	0.020	-	-	-	-
	Maize	0.010	0.002	0.002	-	0.001	-	-
	Cotton	0.003	0.010	0.010	-	0.003	-	-
Moshtohor, Kalyubia	Orange	0.004	0.003	0.030	0.001	-	-	-
	Cotton	0.010	0.001	0.010	-	0.001	-	-
	Cabbage	0.001	0.010	0.010	-	-	6.760	-
	Bean	0.003	0.030	0.003	-	-	-	-
	Maize	0.010	0.010	0.020	-	-	-	-
	Orange	0.050	0.030	0.020	-	-	0.020	-
	Potatoes	0.002	0.010	0.010	-	-	0.030	-
	Carrote	0.002	0.002	0.200	-	-	-	-

- = Not detected.

Table (1): Continued

Localities of samples	Field	Chlorinated residues (ppm)					Organophosphorus residues (ppm)	
		Lindane	Alpha HCH	Total DDT	Dield- rin	Endrin	Malathion	Phospho- methyl
Tanta, Garbia	Orange	0.010	0.002	0.004	-	-	-	-
	Wheat	0.004	0.010	0.010	-	-	-	-
	Vegetable	0.020	0.001	0.060	-	0.006	-	-
	Horticulture	0.007	-	0.002	-	-	0.020	-
El-Amar, Kalyubia	Clover	0.016	0.001	0.009	-	0.006	-	-
	Orange	0.003	0.010	0.02	-	0.002	-	-
Kafer-Farsis, Kalyubia	Vegetable	0.020	0.050	0.050	-	-	-	-
	Cabbage	0.001	0.020	0.001	-	0.001	4.030	-
Tukh, Kalyubia	Cotton	0.010	0.010	0.050	-	0.010	-	-
	Maize	0.003	0.050	0.003	-	-	-	-
	Orange	0.020	0.030	0.450	-	0.001	-	-
	Cotton	0.020	0.020	0.050	0.003	-	-	-
Tamia, Fayoum	Maize	0.020	0.320	0.010	-	-	-	-
	Maize	-	0.020	0.002	-	-	-	-
	Cotton	0.040	0.120	0.002	-	0.001	-	-
	Millet	0.001	0.004	0.001	0.001	-	-	-
Vegetable	Vegetable	0.010	0.070	0.020	0.001	-	-	-
	Cabbage	0.040	0.010	0.020	-	-	-	-

- = Not detected.



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### تقدير متبقيات بعض المبيدات الكلورينية والفوسفورية العضوية في التربة

١٠ د فارس اللقوه ، دامينه محمد خالد ، د٠ علي شمس الدين  
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تم جمع عينات تربيه بالاخص من منطقه الجذر ( ١٥ - ٢٥ سم عمق ) من مناطق مختلفه في جمهوريه مصر العربيه ( كفر الدوار وابو حمص من محافظه البحيره ، محافظه طنطا محافظه بنها ، ( كليه زراعه مشتهر طوخ ) وكذلك مسمم الفيوم لمعرفه مستوي بقايا بعض المبيدات الكلورينية ( اللندان ، المشابه الفاسادس كلوريد البنزين ، د٠ د٠ ت ، الدايلدرين والاندريين ) والفوسفورية العضويه ( الملاثيون والبريميپوس ميثيل )

ولقد اظهرت النتائج احتواء جميع عينات التربه علي بقايا مبيدات كلورنيه وهي اللندان والمشايبه الفاسادس كلوريد البنزين ود٠ د٠ ت٠

احتوت ١٣٩٪ من العينات المختبره علي بقايا المدايلدرين في حين ٢٠٦٪ من العينات احتوت علي بقايا الاندريين ، الا ان كميته كانت ضئيله جدا .

بالنسبه لبقايا المبيدات الفوسفوريه العضويه ، فقمم احتوت ٢٢٢٪ من العينات علي مبيد الملاثيون واظهرت التحليلات احتواء عينتين علي نسب مرتفعه من الملاثيون وقد تطابق هذا مع المعلومات المجمعه والتي تشير الا ان هاتين العينين اخلت من تربيه سبق معاملةها بهذا المبيد منذ فتره قصيره

لم يتم اكتشاف اي اثر لمبيد البريميپوس ميثيل فسمي العينات المختبره