Pakistan J.Sci.Ind.Res., Vol. 27, No. 3, June 1984

RESIDUAL LIFE OF MONOCROTOPHOS, FENITROTHION AND ENDOSULFAN ON COTTON FOLIAGE*

Muhammad Mumtaz; (Miss) Nusrat Nasir; M.J. A. Osmani and M.M.H. Baig

Pesticide Research Laboratory, Pakistan Agricultural Research Council, Malir Halt, Karachi-27

(Received June 19, 1982; revised August 30, 1982)

Cotton crop grown under controlled conditions was treated with concentrates of monocrotophos, fenitrothion and endosulfan at 0.04%, 0.05% and 0.09% active ingredient respectively, in water. Samples of sprayed cotton plants were collected at 0, 1, 2, 4, 7, 15 and 30 days after treatment and analysed for pesticide residues by thin-layer and gas chromatographic techniques.

The residues of monocrotophos were highest (7-10 ppm) in the two day old samples and diminished rapidly thereafter to less than 0.5 ppm in 7 days. No residues were detectable after 15 days. The residues of fenitrothion were highest in zero to one-day old samples (8.5-11.5 ppm) and thereafter decreased to 1 ppm in 7 days, and after 15 days none were detectable.

The residues of endosulfan registered a gradual decrease from 13ppm to 3ppm in zero to seven days old samples, and only 0.2ppm was found after 15 days. No residues were detectable after 30 days.

INTRODUCTION

A nursery of cotton crop was grown in laboratory's premises in order to assess the residues of some of the common pesticides used on cotton crop. Pesticides such as concentrates of monocrotophos, fenitrothion and endosulfan were selected for foliar spray so as to determine pattern of dissipation of these residues. Dilutions of these pesticides in water, at 0.04%, 0.05% and 0.09% active ingredient respectively were sprayed on the cotton crop at an interval of one month in each case.

Two samples of cotton foliage were collected before starting the spraying programme so as to study the recovery of the parent compounds after spiking at 0.2 and 0.5ppm level. Samples for analysis of residue were drawn after 0, 1, 2, 4, 7, 15 and 30 days interval and analysed by standard methods.

MATERIAL

i) Monocrotophos 40% water soluble concentrate obtained from Pakistan Burmah Shell.

ii) Fenitrothion 50% emulsifiable concentrate (Sumitomo, Japan).

iii) Endosulfan 35% Emulsifiable concentrate (Hoechst, West Germany).

iv) All solvents used in the present study were of Reagent Grade. Methanol and n-hexane were double distilled.

v) Thin-Layer chromatography, TLC sheet Woelm silica gel; Aluminum sheets 20 x 20 cm, thickness of Aluminum sheet support was 0.1 mm with silica gel layer of 0.2 mm thickness. Chromogenic agent for TLC was 2% Nitro benzylpyridine and 10% tetraethyl pentamine in acetone.

vi) Gas chromatography apparatus, Pye-Unicam Panchromatograph.

vii) Glass Column (1.8 x 40 cm) with stopcock was used for column chromatographic clean-up of crop extracts using Florisil, BDH, 100 mesh as the absorbent.

METHODS

a) Extraction for Monocrotophos Residues. A representative portion of the samples were reduced into small pieces with the help of a cutter. 50 ± 0.5 g of finely divided material was transferred into a blender with 100-125 ml of methanol. The sample was thoroughly macerated (3-4 minutes were sufficient). The contents were allowed to stand for 5 minutes followed by decantation of the mobile extract and filtering through a 7cm Buchner funnel fitted with grade 541 filter paper into a 250 ml graduated cylinder. The crop matrix was quantitatively transferred with several 30 ml portions of methanol until 250 ml of filtrate was obtained. The extract was thoroughly mixed and transferred to a clean dry bottle. The extract represented 1g of

*Investigations conduced from funds provided under Project No. PK-ARS-137 (Gratn No. FG-Pa-299).

crop per 5 ml of extract and assayed for monocrotophos residues by thin-layer chromatography.

b) Extraction and Clean-up for the Determination of Fenitrothion Residues. To the 50 g sample was added 50 ml of water and 25 ml of methanol with blending at high speed for 30 seconds. It was followed by the addition of 100 ml acetonitrile and blending for one minute. The crop matrix was then filtered using a Buchner funnel. The filtered cake was again blended with 50 ml mixture of water/methanol/acetonitrile (2:1:4) and filtered as above. The combined filterate was shaken with 30 g of sodium chloride and 150 ml of chloroform for 5 minutes. The lower chloroform layer was separated and the aqueous phase was shaken with an additional 100 ml of chloroform and the chloroform layer was obtained. The combined chloroform layer was dehydrated with 50 g; of anhydrous sodium sulphate by filteration followed with 50 ml chloroform rinsing. The chloroform extract so obtained was concentrated at 40°C under vacuum using water pump. The concentrated extract was taken-up in 20 ml of n-hexane saturated with acetonitrile and 20 ml of acetonitrile saturated with n-hexane. The whole extract was shaken for 10 minutes and then the lower acetonitrile phase was obtained. The n-hexane phase was shaken for 5 minutes with an additional 10 ml of acetonitrile saturated with n-hexane and the acetonitrile layer was separated. The combined acetonitrile extract was then concentrated just to dryness using a rotary evaporator and taken up in n-hexane (5 ml) for monitoring residues of fenitrothion by gas chromatography with electron capture detector.

c) Extraction and Clean-up for Endosulfan Residues. 50 g crop sample was blended with 10 g of sodium sulphate. 50 ml of solvent mixture n-hexane/isopropyl alcohol in the ratio of 2:1 was added and blended at high speed for 30 seconds. The plant matrix was blended 3 times at small intervals. The blended material was filtered through the Buchner funnel as usual and the filtrate was concentrated. The concentrated extract was taken up with 10 ml of n-hexane and passed through the glass column containing 10 g florisil topped with 5-0 g of anhydrous sodium sulphate. The column was eluted with (30:70) mixture of ethyl ether and petroleum ether. The eluate obtained from the column was concentrated on a rotary evaporator just to dryness. The concentrated extract was then taken up in 5 ml n-hexane for monitoring residues of endosulfan by gas chromatography with electron capture detector.

Table 1. Residues of parent monocrotophos, fenitrothion and endosulfan after foliar spray on cotton plants.

Sample interval after spray in days	ppm monocrotophos by TLC	ppm fenitrothion by GLC	ppm endosulfan by GLC
0	8-10	11.5	13.0
1	8	8.5	12.0
2	7	5.0	8.0
4	2	5.0	2.0
7	0.5	1.0	3.0
15	N.D.	N.D.	0.2-0.5
30	N.D.	N.D.	N.D.

Results of residues found by TLC and GLC are given in Table 1.

CONCLUSION

This work indicates that:

1. The residues of monocrotophos were highest (7-10 ppm) upto 2 days samples and diminshed thereafter to less than 0.5 ppm in 7 days.
2. The residues of fenitrothion were highest in 0-1 days sample (8.5-11.5 ppm) and thereafter diminished to 1 ppm in 7 days and after 15 days none were detectable.
3. The residues of endosulfan registered a gradual decrease from 13 ppm to 3 ppm in 0-7 days and only 0.2 ppm was found after 15 days. No residues were detectable after 30 days.

REFERENCES

1. Proper Selection and correct use of Pesticides for Cotton in Pakistan, Central Cotton Institute, Multan (June, 1980).
2. D.E. Coffin, and G. Savary, J. Assoc. Official Anal. Chemist, **47**, 875 (1964).
3. A. Luke Milton, *et al.* Journal of the AOAC, **58**, 1020 (1975).
4. Analytical Methods for Pesticides, Plant Growth Regulators and Food Additives, Gunter Zweig, Vol. VI.
5. Analytical Methods for Pesticides and Plant Growth Regulators. Vol. VII. Thin-layer and Liquid Chromatography and Analysis of Pesticides of International Importance.