

DISSIPATION OF ENDOSULFAN IN COWPEA

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Abstract : In an experiment to study the dissipation of endosulfan in cowpea pods following application @ 0.07 and 0.14 per cent at flowering and pod formation stage, the residues dissipated below detectable level on 15th day in the lower dose while 0.3 mg kg⁻¹ could be detected in the higher dose. A waiting period of 2.85 and 5.21 days are fixed for the lower and higher dose respectively. Washing of green pods removed 39.78% of the initial deposit while washing followed by cooking removed up to 67.93% in the lower dose.

Key words : Cowpea, decontamination, endosulfan residues.

INTRODUCTION

Cowpea, *Vigna unguiculata* L. is affected by a spectrum of pests in the flowering and pod formation stages. To achieve an effective control of pod borers and leaf miner, farmers regularly use systemic insecticides at an interval of 3 to 4 days. Safer and effective insecticides are to be identified for the management of pod borers with a view of minimising harvest time residues in cowpea pods. Hence an experiment was conducted to study the dissipation of endosulfan for which the maximum residue limit (MRL) prescribed by FAO and WHO is relatively higher.

MATERIALS AND METHODS

A field experiment was laid out in plots of 5 m² at the Instructional Farm, College of Agriculture, Vellayani, Trivandrum during July-September 1992 using the cowpea variety C-152. Endosulfan 0.07% (500 g ai ha⁻¹) and 0.14% (1000 g ai ha⁻¹) were applied @ 700 l ha⁻¹ at the flowering stage and 25 days later at the pod formation stage. Pod samples were drawn randomly at 0 (2h), 1, 3, 5, 7, 10 and 15 days after the second spraying. Grain and pod cover were collected at the first harvest. The mean temperature during the first application of insecticide and the last sampling ranged from 23.2°C to 30°C and the percentage relative humidity from 88 to 96. The average sunshine hours during the period was 8.8 and total rainfall was 62.6 mm.

A representative of fresh pod sample (50 g) was taken for analysis. The pod samples were

extracted twice with 150ml mixture of isopropanol and n-hexane (1:2 V/V) by blending each time for 3 min. The extract was diluted with 100 ml distilled water to remove the aqueous layer and the n-hexane extract was dried over sodium sulphate. The n-hexane extract was concentrated, and dissolved in 45 ml n-hexane and 5 ml of acetone. Darco-G 60 (0.5 g) was added and allowed to stand for 1 min with occasional shaking. The mixture was filtered through filter paper (Whatman No.1) and washed the residue thrice with 15 ml n-hexane : acetone (9:1) mixture. The extract was again concentrated and dissolved in distilled hexane to inject in GC.

The grain and the dry pod cover after grinding to coarse powder were extracted in a soxhlet apparatus using n-hexane as the solvent. The materials were refluxed over a water bath at 80°C for 6 hours. The extracts were cleaned up by the acetonitrile partitioning technique (Jones and Riddich, 1952). The residues of the metabolites viz., alpha, beta, and endosulfan sulphate were determined by gas chromatography using Chemito Model 3865 equipped with electron capture detector and 2 m x 2 mm glass column packed with 3 per cent OC 17. The column, injection and detector temperature were 200°C, 220°C and 250°C respectively and the flow of the carrier gas N₂ was 80 ml min⁻¹.

The efficiency of extraction, clean up and estimation procedure was checked by recovery experiments for endosulfan by fortifying the respective samples. The recovery values of pods, grains and pod cover ranged from 80 to 90 per cent.

Pod samples of 0 day (2 hours after application) and 7th day were subjected to washing alone and washing followed by cooking to assess the effect of processing on the removal of the residues. The rate of dissipation of endosulfan residues was worked out by determining RL_{50} values. T_{MRI} values were statistically worked out to obtain the waiting period following the method of Hoskins (1961).

RESULTS AND DISCUSSION

The residue data of endosulfan on cowpea pods at different sampling intervals are given in Table 1. A perusal of the data revealed that the initial deposit of endosulfan on green pods

of cowpea after 2 hours of spraying was 3.72 and 6.30 $mg\ kg^{-1}$ for the doses of 0.07 and 0.14% respectively. The residues in the pods after 1, 3, 5, 7 and 10 days of second application were 2.95, 2.04, 1.08, 0.69 and 0.09 $mg\ kg^{-1}$ respectively when applied @ 0.07% spray. The corresponding values were 5.14, 3.98, 1.96, 1.03 and 0.60 $mg\ kg^{-1}$ respectively when applied @ 0.14%. No detectable residue was seen on 15th day of application for the lower dose while 0.31 $mg\ kg^{-1}$ was observed for the higher dose. The reduction in the level of residues in the days following application may be attributed to physical removal by the weathering agencies as explained by Gunther and Blin (1955).

Table 1. Mean residues of endosulfan on cowpea pods ($mg\ kg^{-1}$)

Sampling interval, day	Endosulfan 0.07%				Endosulfan 0.14 %			
	alpha	beta	Sulphate	Total	alpha	beta	Sulphate	Total
0 (hr)	2.86 + 0.222	0.86 ± 0.102	ND	3.72	4.86 ± 0.467	1.44 ± 0.142	ND	6.30
1	2.21 t 0.437	0.74 ± 0.058	ND	2.95	3.81 ± 0.413	1.53 ± 0.108	ND	5.14
3	1.06 t 0.173	0.62 ± 0.120	0.36 ± 0.049	2.04	2.37 ± 0.113	1.06 i 0.009	0.55 ± 0.080	3.98
5	0.58 + 0.078	0.34 ± 0.056	0.16 t 0.041	1.08	0.87 ± 0.174	0.79 ± 0.078	0.30 ± 0.030	1.96
7	0.32 i 0.039	0.30 t 0.049	0.07 ± 0.009	0.69	0.41 ± 0.053	0.53 ± 0.086	0.09 ± 0.024	1.03
10	0.04 ± 0.009	0.06 ± 0.019	0.01 ± 0.00	0.09	0.23 ± 0.057	0.30 ± 0.65	0.07 t 0.016	0.60
15	ND	ND	ND	ND	0.14 ± 0.032	0.11 ± 0.020	6.60 ± 0.016	0.31
Reg. equation $y = 1.106 - 0.2821x$					$y = 0.7837 - 0.0931x$			
T tol (days) 2.85					T tol (days) 5.21			

The initial deposits of alpha and beta endosulfan were 2.86 and 0.86 $mg\ kg^{-1}$ respectively for lower dose while it was 4.86 and 1.44 $mg\ kg^{-1}$ for the higher dose. Endosulfan sulphate was detected from 3rd

day onwards and the percentage isomer ratio of alpha endosulfan, beta endosulfan and endosulfan sulphate on the third day was 52:30:18. The waiting period of endosulfan in cowpea pods when treated at 0.07% was 2.85

Table 2. Effect of decontamination techniques on the removal of endosulfan residues (mg kg⁻¹) in cowpea

Sampling stage, day	Unwashed				Washed				% removal	Washed & cooked				% removal
	alpha	beta	sulphate	Total	alpha	beta	sulphate	Total		alpha	beta	sulphate	Total	
A. Endosulfan 0.07%														
0	2.86	0.86		3.72	1.78	0.46		2.24	39.78	1.02	0.41		1.48	60.21
7	0.32	0.30	0.07	0.69	0.25	0.24	0.02	0.51	26.09	0.19	0.13	0.01	0.33	52.17
B. Endosulfan 0.14%														
0	4.86	1.44		6.30	2.03	1.75	-	3.78	40.00	1.81	0.52		2.33	63.02
7	0.41	0.53	0.09	1.03	0.25	0.28	0.07	0.60	41.74	0.18	0.20	0.03	0.41	60.19

days and at 0.14% was 5.21 days. These results are in conformity with the findings of Rampal and Handa (1994) who reported that endosulfan residues in soybean pods reached below the MRL of 2 mg kg⁻¹ as fixed by FAO (1985) in 3.10 days following application @ 0.07% at the pod formation stage.

The data pertaining to the effect of the decontamination process are presented in Table 2. Washing of 0 days (2 hours) cowpea pods for one minute under water led to the removal of 39.78 and 40.00% of the residues from the normal and double dose respectively. But there was remarkable reduction of 60.21 and 63.02% of residues when (2 hours) pods from normal and double dose were subjected to washing followed by cooking. The corresponding values for the 7th day samples were 52.17 and 60.19%. These findings are in conformity with those of Rampal and Handa (1994) who observed that washing in water and cooking decreased endosulfan residues by 60-86% in soybean. Thus decontamination processes render pods safer for consumption by reducing the residues.

Thus based on the harvest time residue data, endosulfan can be identified as a safe insecticide for the insecticidal management of pod borers. Field evaluation may be taken up to assess the bioefficacy of endosulfan in comparison with the presently recommended insecticides.

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