https://doi.org/10.29321/MAJ.10.A02829

Madras agric J. 67 (4) : 256–260, April 1980

Dissipation of Monocrotophos Residues in Black gram Husk and Grains

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The residue of monocrotophos in husk and grains of black gram was analysed by harvesting at two different periods, (12 and 20 days) after spraying both by colorimetry and bio-assay methods. The residues of monocrotophcs were at non-detectable levels in husk even on 12th day after spraying. The residues in grain were below 0.2 ppm which is the tolerance limit for beans when assayed chemically at both periods. However, ths residues were slightly above the tolerance limit both at 12 days and 20 days after spraying when determined by bio-assay. The study indicated that a minimum waiting period of 20 days is necessary for safe consumption.

Blackgram (*Vigna mungo*) is an impotant pulse crop extensively grown in Tamil Nadu. A number of insect pests infest black gram and among them, damage due to stem fly *Ophiomyia phaseoli* Tryon and pod borers are very high (Anon. 1971). Monocrotophos at 0.03% in 800 litres/ha was recommended for the control of stem fly in legume vegetables (Ootwani and Butani, 1977). Chelliah *et al.* (1977) recommended application of aldicarb, phorate or disulfoton granules at 1.0 Kg a.i./ha at seeding followed by spraying with monocrotophos at 0.04% at flowering for black grom pod borers. As some blackgram varieties are harvested in 65-70 days there is the possibility of the presence of toxic residues of monocrotophos in grains which may be hazardous, if consumed. Hence to find out the possible amount of residues of monocrophos sprayed on black gram plants and to fix the waiting period before harvest, this experiment was conducted under field conditions.

MATERIALS AND METHODS

A field experiment was conducted at Tamil Nadu Agricultural University Coimbatore, during April-July, 1977, in randomised block design with ten treatments replicated three times to test the efficacy of certain insecticides in the control of pests of blackgram with C0.2 variety. Among the treatments monocrotophos (Nuvacron) was sprayed on blackgram plants at the rate of 0.4 Kg. a.i./ha on 7th, 21st and 35th days after sowing, It was also sprayed on 55th day especially for the control of pod borers. Composite samples of pods, the first sample on 12 days after final spraying and second sample on 20 days after final spraying, were harvested and taken

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* Part of M. Sc.(Ag) Thesis sudmitted by the first author to Tamil Nadu Agricultural University, Coimbatore-3

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for analysis. The residues were anaysed both in husk and grains byl both chemical and bio-assay methods.

CHEMICAL ASSAY:

The chemical assay of monocrotophos residues in the husk and grains was estimated following the method of Getz and Watts (1964).

Fifty g of the composite sample of both husk and grain was blended with 100 ml of acetone for three minutes and the contents were filtered through Buchener funnel. The extracted husk and grains were respectively reextracted by blending with 50 ml of acetone and filtered. The filtrate was concentrated in Kuderna - Danish evaparator to 10 ml and was transferred to a 500 ml separating funnel by washing with 5 ml chloroform. Then 100 ml distilled water and 10 to 25 ml saturated NaCl solution were added and the valume made up to 200 ml with chloroform. The separating funnel was shaken thoroughly for one minute and allowed to separate.

The lower layer of chloroform was passed through a funnel containing anhydrous sodium sulphate over glass wool. The aqueous phase in the separating funnel was extracted with fresh 10 to 15 ml chloroform and the above process was repeated. The volume was made upto 50 ml. An absorption mixture of activated charcoal, magnesium oxide and celite in the ratio of 1:1:1 respectively was prepared and taken in a chromatographic column and the extract was added with five g of activated charcoal and passed through the column. The colourless filtrate collected was made up to 50 ml with chloroform.

Clean test tubes were taken and 5, 10, 15, 20 and 25 ug of monocrotophos was added and the volume was made equal in each test tube by adding required amount of solvent.

The solvent was evaporated in an air condenser. Then 0.4 ml of 2% solution of (p-nitrobenzyl) pyridine in acetone and 0.4 ml of 2% solution of cyclohexylamine solution in acetone were added. Each tube was heated for three minutes in an oil bath maintained at 170°C (The oil bath was kept at 200°-210°C so that when the tubes were put the temperature became 170.-180.C). Then the test tubes were dipped in an ice bath and 3 ml of ethyl acetate was added to each tube and the absorption read at 540 mu. The absorbance vs. concentration was plotted in a graph to obtain a straigt line curve.

Known volume of elute was taken in a standard joint test tube. The solvent was removed usnig kuderna-Danish evaporator and the procedure outlined earlier was followed. The concentration was determined by plotting in the standard curve and by multiplying the factor, the amount of residue was worked out.

The method suggested by sun *et al* (1965) was used to construct standard

curvefor bio-assay. One day old unsexed *Drosophila melanogaster* Meig., flies were used as test insect Exactly 0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 ug of monocrotophos in acetone were added separately to well cleaned 100 ml beakers and solvent was evaporated with a gentle rotation of rhe beakers against an air current. Five minutes after the complete evaporation of the solvent 25 flies were introduced. The beakers were covered with cheese cloth and secured with rubber bands The dead and moribund flies were counted after 5 hours and dosage mortality curve was prepared for monocrotophos based on log concentrations and probit values of mortality. One ml of the residue extract was taken per beaker and the mortality percentage of *Drosophila* flies was calculated. The quantity of monocrotophos was ascertained from the standard curve and was converted to parts per million.

Samples of husk and grains were analysed for residue using the procedure for chemical assay to calculate the percentage of recovery.

RESULTS AND DISCUSSION

The recovery percentage through the method described and the residue levels in husk and grains of blackgram by chemical and bio-assay methods a different days of harvest are presented in Table I and II.

The results indicate that the method adopted for estimation of monocrotophos in blackgram is a reliable one as the recovery perentage by chemical assay is 84.60 and 88.30 in husk and grains respectively.

The residues were not detectable in husk when assayed on 12th as well as 20th day after final spraying by both chemical and bio-assay methods in grains, however the residues were 0.18 and 0.05 ppm by chemical assay and 0.33 and 0.27 ppm by bio-assay respectively on 12th and 20th day after spraying. As there is no information on the residues of monocrotophoson blackgram, results on cowpea is being compared. Awasthi *et al.* (1977) reported that the residues of 0.03 and 0.05% monocrotophos treatments at 10 days after the spray on cowpea pods were 0.22 and 0.58 ppm respectively. After post-repeat application, the residues were not detectable and 0.09ppm respectively at 15 days after application They also reported that the with holding peroid for monocrotophos used at 0.75, 1.25 and 3.12 kg/ha on cowpea was 11-15 days.

The tolerance limit for this chemical for beans is 0.2 ppm (Anon.,1976 In chemical assay, the residue was below the tolerance limit even on 12th day after final spraying and reduced further on 20th day after spraying But the residues were slightly above the tolerance limit when tested even on 20th day after spraying by bio-assay indicating that a waiting period of 20 days has to be allowed.

The senior author wishes to acknowledge M/s. Ciba-Geigy (India) Ltd., for financial assistance.

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TABLE I Recovery from fortified sample of monocrotophos (Chemical assay)

	HUSK		GRAINS	
Fortified (ppm)	Recovery (ppm)	Recovery (Percentage)	Recovery (ppm)	Recovery (Percentage)
0	0	0	0	0
2	1.62	81.00	1.70	85.00
5	4.25	85.00	4.55	91.00
10	8.80	88.00	8.90	89.00
Mean		84.60		88.30

TABLE II Monocrotophos residue in black gram husk and grains (Mean of three readings)

Sl. No.	Days after final spraying	Total insecticide input	Residue in ppm				FAO/WHO tolerance limit for beans in ppm
			Chemical assay		Bioassay		
			Husk	Grain	Husk	Grains	
1.	12	0.4 Kg	N. D.	0.18	N. D.	0.33	0.2
	20	ai/ha	N. D.	0.05	N. D.	0.27	