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Determination of Endosulfan Residue on/in Brinjal Fruits

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ABSTRACT

Chemical assay of endosulfan residue was determined by spraying of two different concentrations as well as different number of application. The residue of endosulfan on the brinjal fruit at the lower concentration (0.05 per cent) did not reach below tolerance level even after 15 days in case of three sprayings of endosulfan after washing the fruits. There was reduction in residue content below the tolerance level after 10 days of the treatment of one spraying of 0.075 per cent endosulfan when the fruits were washed with tap water.

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INTRODUCTION

Endosulfan is one of the most common insecticides being used for the control of fruit borer of brinjal crop. Ganguli *et al.*, (1974), Purohit and Khatri (1975) as well as Shah (1979) reported endosuflan being good for the control of the fruit borer.

Since endosulfan is the most common insecticide being used in the brinjal field of South Gujarat, it was thought imperative to investigate through this research the status of residue of endosulfan on/in brinjal fruit due to its repeated use as a part of plant protection measures, against brinjal fruit borer under the South Gujarat conditions so as to fill the lacunae of information in our knowledge regarding its residue level as well as its waiting period. This information will be of much use to organise plant protection practices not only for the effective control of an insect pest but also from the point of view of hazards to the consumers.

MATERIALS AND METHODS

Technical grade of endosulfan was used for preparing standards caliberation curve using distilled n-hexane

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(Figure-1). The commercial grade of endosulfan 35 per cent EC of M/s Hoechst Pharmaceutical Limited, Bombay was used for spraying brinjal crop.

An experiment was carried out at the farm of N. M. College of Agriculture, Navsari during 1978-79 in randomised block design with three treatments and four replications. Treatments consisted of :

(1) Endosulfan 0.05% three sprayings at an interval of 21 days starting from initiation of flowering.

(2) Endosulfan 0.075 % three sprayings at an interval of 21 days starting from initiation of flowering.

(3) Endosulfan 0.075% one spraying after 42 days of initiation of flowering.

The calorimetric method employed for endosulfan residue analysis was one that was based on method of Bulter *et al.*, (1962) and modified by Maitlen *et al.*, (1963). The cleanup technique modified by Kathpal and Dewan (1975) was employed in this research.

The brinjal fruits were plucked randomly from each treatment plot and immediately transferred to polythene bags duly tagged and perforated for aeration. The samples were harvested after last spraying at an interval of 0, 3, 7, 10 and 15 days. For estimation of endosulfan residue on the brinjal fruits, one brinjal fruit was taken from each sample, weighed and put in a one litre glass jar and washed with 100 ml. n-hexane for 10-15 minutes. The aliquot was then filtered through a funnel over anhydrous sodium sulphate. It was then concentrated to 50 ml by way of keeping on water bath. This solution was then transferred into a labelled air tight bottle and preserved in a refrigerator until further use.

For the estimation of the endosulfan residue in the brinjal fruits (i.e. washing treatment) one brinjal fruit from each sample was washed with tap water for half minute and allowed to dry. It was chopped to a size of 0.5 to 1 cm. with knife. Forty gm of the chopped fruits were then taken and put into blender and blended for 1.5 minutes by adding 80 ml. of n-hexan-is propanol mixture (2:1). The extract was filtered through a Buchaner funnel by using a suction pump. It was then washed 3 times with 25 ml of 5 per cent aqueous sodium sulphate solution. For removal of isopropanol, the filterate was then transferred into a separating funnel and shaken vigorously for 2 to 3 minutes and allowed to stand for 5 to 10 minutes for separating n-hexane layer. The upper layer was taken out discarding the lower aqueous layer. The n-hexane extract was filtered through 2-3 inch column of anhydrous sodium sulphate. The aliquot was concentrated to a volume of 40 ml. and then transferred into a bottle and preserved in a refrigerator for further use.

Cleanup was done by adding 10 ml. of acetone and 1 gm decolourizing carbon Nuchar-c-190-N in beaker and shaken for about one minute and allowed to stand for two minutes to settle the absorbant. The supernatant solution was then filtered through anhydrouls sodium sulphate. Beaker and funnel were washed three times with 15 ml.

of fresh solvent mixture of n-hexane and acetone (80:20). Finally, the beaker and anhydrous sodium sulphate layer were rinsed with the above 20 ml. of solvent mixture. The filterate solution was concentrated to a quantity of 5 ml. and it was transferred into a stoppered test tube.

One drop of mineral oil was added into each test tube with the help of a dropper, then these test tubes were put into a hot water bath at 50° C to dryness. Finally, 7.5 ml. of freshly prepared methanolic sodium hydroxide pyridine reagent was pipetted into each test tube and they were then stoppered, and duly tied with rubber bands. These tubes were immersed for four minutes in oil bath at 100 ± 2^0 C, then subsequently kept in ice water bath for one minute. Stoppers were removed from test tubes. Absorbance of colour complex developed, was measured in spectronic-20 at 520 mμ against distilled water.

RESULTS AND DISCUSSION

Three sprayings of endosulfan were done at the rate of 0.075% and 0.05% on brinjal crop at an interval of 21 days. The initial deposit of endosulfan residue on fruit immediately after last spraying was 69.84 ppm, and the residue after 3rd, 7th, 10th and 15th days was 25.78, 19.18, 15.28 and 10.89 ppm respectively in case of 0.075% endosulfan (Table 1). Thus the percentage reduction of endosulfan residue was 63.09, 72.54, 78.12 and 84.41 per cent after 3rd, 7th, 10th and 15th day respectively after last spraying of endosulfan.

The initial deposit of endosulfan residue in case of three sprayings of 0.05% was 52.16 ppm and residue after 3rd, 7th, 10th and 15th days was 18.20, 15.76, 11.12 and 7.25 ppm respectively. The percentage reduction of endosulfan residue was 65.11, 69.79, 78.68 and 86.10 per cent (Table 1). Similarly only one spraying of endosulfan 0.075%

TABLE 1

Residue of endosulfan on/in brinjal fruits by chemical assay.

Stage of sampling	*Three spraying*								*One spraying*			
	0,075%				*0.05%*				*0.075%*			
	Residue in ppm		*Percentage of reduction*		*Residue in ppm*		*Percentage of reduction*		*Residue in ppm*		*Percentage of reduction*	
	on	*in*	*on*	*in*	*on*	*in*	*on*	*in*	*on*	*in*	*on*	*in*
1. Immediately after last spraying	69.84	26.89	—	—	52.16	20.38	—	—	66.24	6.86	—	—
2. 3 days after the last spraying	25.78	10.33	63.09	61.58	18.20	6.56	65.11	58.00	22.66	3.46	65.79	49.56
7 days after the last spraying	31.9.18	7.94	72.54	70.47	15.76	6.10	69.79	70.07	16.20	2.16	75.54	68.51
4. 10 days after the last spraying	15.28	5.12	78.12	80.96	11.12	3.45	78.68	83.07	12.96	0.75	80.44	89.07
5. 15 days after the last spraying	10.89	2.56	84.41	90.48	7.25	2.09	86.10	89.79	8.24	BDL	87.56	100.

was given at the same time, when third spraying of endosulfan 0.05% and 0.075% were done. The estimation of endosulfan residue was then done and the results obtained are presented in Table 1. The initial deposit of endosulfan residue on fruit was 66.24 ppm while the residue was 22.66, 16.20, 12.96 and 8.24 ppm after 3rd, 7th, 10th and 15th days of spraying respectively. Thus the percentage of reduction of endosulfan residue was 65.79, 75.54, 80.44 and 87.56 per cent after 3rd, 7th, 10th and 15th day of spraying of endosulfan respectively.

The initial deposit of endosulfan residue in the fruits i.e. after half minute washing of fruits with tap water was 26.89 ppm and the same after 3rd, 7th, 10th and 15th day of last spraying of endosulfan were 10.33, 7.94, 5.12 and 2.56 ppm respectively in case of three spraying of 0.075% endosulfan while it was 20.38 ppm as initial deposit but it was 6.56, 6.10, 3.45 and 2.09 ppm after 3rd, 7th, 10th and 15th day of last spraying respectively in case of three sprayings of 0.05% endosulfan (Table 1). Similarly in case of one spraying of endosulfan 0.075% the initial deposit in the fruits was 6.86 ppm whereas residue in the fruits after 3rd, 7th, 10th and 15th day of spraying was 3.46, 2.16, 0.75 and BDL respectively (Table 1).

The reduction of residue in brinjal fruits was 61.58, 70.47, 80.96 and 90.48 per cent for endosulfan 0.075% whereas it was 58.00, 70.07, 83.07 and 89.79 per cent for endosulfan 0.05% on 3rd, 7th, 10th and 15th day respectively after last spraying. In case of one spraying of endosulfan 0.075% the reduction of endosulfan residue was 49.56, 68.51, 89.07 and 100.00 per cent on 3rd, 7th, 10th and 15ht day of the spraying respectively.

From the above results, it can be inferred that there was much difference in initial deposit of endosulfan arising due to high and low concentration. But the residue reduction percentage was almost same in both the concentrations. Leitao and Fernandes (1966) reported that the endosulfan sprayed at the rate of 52.5 and 105 ml. a.i./ha in the tomato fruits showed a rapid reduction of its residue in first 4 days. Benoit (1973) found that endosulfan residue reached below the tolerance level in 34 days in lettuce. Srivastava and Kavadia (1976) reported that 3 sprays of 0.1% endosulfan on fig fruits took 30 days to reach below the tolerance level. Valand and Shah (1978) found that the endosulfan residue of 5.66 ppm and 2.53 ppm i.e. above tolerance limits existed after 25/19 days in mango fruit. It is thus evident from the above reports that the endosulfan may take such a long period of time of about 30 days for reaching below the tolerance level of 2 ppm as prescribed by FAO/WHO recommendations as reported by Anonymous (1972). This reports support the present findings where residue of endosulfan on brinjal fruits did not reach below tolerance level even after 15 days of last spraying.

The dissipation of the endosulfan residue attained below the tolerance level of 2 ppm after 10 days in case of one spraying of 0.075% concentration, while the residue did not reach below the tolerance level even after

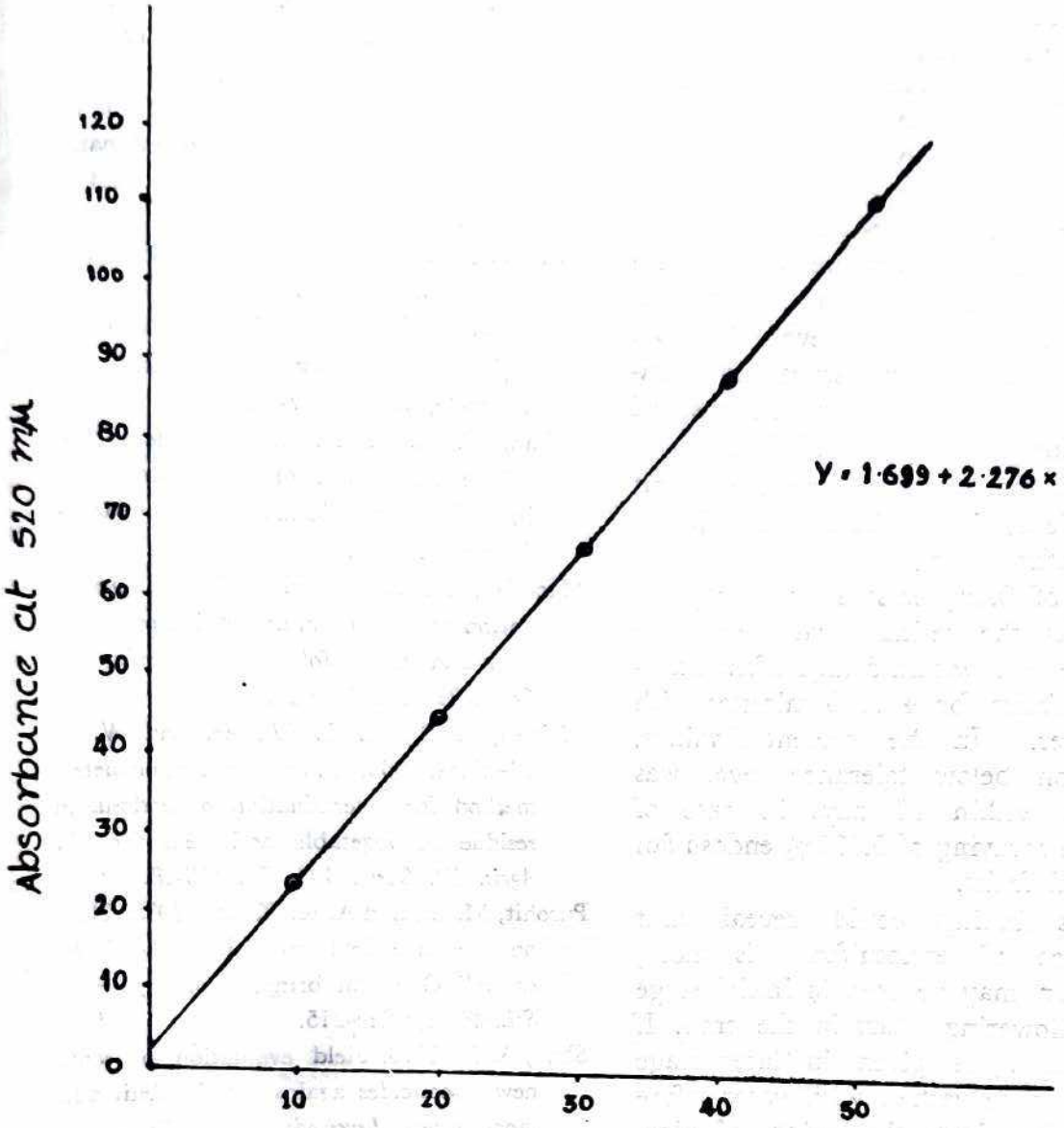

Fig.1 Endosulfan concentration in microgram

Standard Caliberation Curve For Endosulfan By Chemical Assay

15 days in case of three sprayings of endosulfan after washing the fruits. Valand and Shah (1978) found that the dissipation of the endosulfan residue attained below tolerance level after 7 days in case of only one spraying and after 25 days in case of seven sprayings in mango fruits. Mac-Neli and Hikichi (1976) found that residue of pear tree (6 lb of thiodan 50 wp. per acre) were below the tolerance level of 2 ppm after fourteen days. Srivastava and Kavadia (1976) reported that in case of 0.1% endosulfan spray dissipation of endosulfan below the tolerance limit was achieved in fig fruit within fifteen days. Kathpal *et al.* (1977) found that in case of 0.1% endosulfan spray on ber fruits the residue was below the tolerance level within 5 days after washing the fruits for 4 to 5 minutes with tap water. In the present findings, dissipation below tolerance level was achieved within 10 days in case of only one spraying of 0.075% endosulfan in brinjal fruits.

This findings could reveal that persistence of endosulfan is more, endosulfan may be used in initial stage i.e. till flowering start in the crop. If one spraying is given in later stage fruit should be washed with water before consumption from the point of view of human hazard.

REFERENCES

Anonymous, 1972, Pesticide residue in food F.A.O. agricultural studies, No. 88, F.A.O. Rome.

Benoit, F. 1973. The break-down of endosulfan. *Tuin bouwberichten* **37** (8) : 261-264.

Butler, L. I., J. C. Maitlen and J. E. Fehey 1962. Micro determination of thiodan residue. *J. Agric. Fd. Chem.* **10** (6): 479.

Ganguli, R. N., D. R. Roy and A. K. Roychaudhury. 1974. Control of brinjal fruit and shoot borer in Tripura. *Pl. Prot. Bull. India,* **23** (2): 25-28.

Kathpal, T. S. and R. S. Dewan, 1975. Improved clean up technique for estimation of endosulfan and endrin residue. *J. Assos. offi. Anal. Chem.,* **58** (5): 1076-1079.

Kathpal, T. S. and V. S. Bhatnagar, H. C. L. Gupta and B. P. Srivastava, 1977. Residue of endosulfan in/on ber (*Zizyphus jujuba* Lank) fruit and leaves. Pesticide, **11** (8): 37-39.

Naitao, T. A. and A. M. Fernandes, 1966. The break down of the organochlorine inecticides (Endoslulfan) in tomatotoes *Frutoes.* 119-125.

Mac-Neli, J. D. .and M. Hikichi, 1976. Degradation of endosulfan and ethion on pears, pear and grapes foliage. *J. Agric. Fd. Chem.* **24** (3): 608-611.

Maitlen, J. C., K. C. Walker, and W. E. Westlake. 1963. An improved calorimetric method for determination of endosulfan residue in vegetable and beaf fat. *J. Agric. Fd. Chem.* **111** (5): 416-18.

Purohit, M. L., and A. K. Khatri 1975. Note on the chemical control of *Leucinodes orbonalis* Guen. in brinjal *Indian J. Agri. Sci.* **43** (2): 214-215.

Shah, A. H. 1979. Field evaluation of some new insecticides against brinjal fruit and shoot borer. *Leucinodes orbonalis* Guen. *Indian J. Ento.,* Vol. 4 (2), pp. 195-196.

Srivastava, A. S., and V. S. Kavadia, 1976. Residue of endosulfan in/on fig fruits. Abstract paper of all India Symposium on modern concept in plant protection at University of Udaipur , March 26-28, 1976, p. 75.

Valand, V. M. and A. H. Shah 1978. Determination of residue of carbaryl and endosulfan on/in mango fruits. A thesis submitted to Gujarat Agricultural University, Dantiwada.