

## Bioefficacy and Residue of Endosulfan 50 WDG in Mango

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### Abstract

Studies were conducted to evaluate the efficacy of endosulfan 50 WDG against mango hoppers (*Amritodus atkinsoni* Leth., *Idioscopus niveosparus* Leth. and *Idioscopus clypeales* Leth.) along with carbaryl and nimbecidine on cv *Himsagar* under field condition during 2004-05. Endosulfan at 0.07% (single dose) and 0.14% (double dose), carbaryl at 0.15% and nimbecidine (0.2%) were applied. In total there are five treatments including control. The insecticides were applied at fortnight intervals starting from the panicle emergence. Considering the overall efficacy endosulfan was found to be superior in controlling the hopper population over other insecticidal treatments with more than 80% avoidable yield loss. Dissipation of endosulfan was also studied in mango fruits. The residues declined to non-detectable levels on 60 day after last spray for both the treatment doses. The corresponding half-life was found to be 2.73 day for single dose and 2.95 day for double dose. The safe waiting periods ( $T_{MRL}$ ) determined for endosulfan was 14–20 days. Harvested fruits had no detectable level of residues.

**Key words :** Endosulfan, Bioefficacy, Residue, Mango.

Three species of idiocerine leaf hoppers namely *Amritodus atkinsoni*, *I. clypealis* and *I. niveosparus* are commonly found infesting mango under West Bengal agroclimatic condition causing a loss of 20–100% of inflorescence (1). However, in spite of adopting the recommended control measures, very often severe damage is caused by the pest. To overcome this problem recently introduced newer formulation of endosulfan (50 WDG) has been tested along with carbaryl (0.15%) and nimbecidine (0.2%, 300 ppm). Residue of newer formulation of endosulfan in mango fruits was also studied to evaluate safe waiting period.

### Methods

The statistically designed field experiment was conducted at Mondouri, BCKV, Nadia, WB during 2004-05. A total 15 trees of mango cv *Himsagar* (10 years old) were selected for this experiment. Treatments were endosulfan at 0.07% (single dose,  $T_1$ ) and 0.14% (double dose,  $T_2$ ), carbaryl at 0.15% ( $T_3$ ) 300 ppm nimbecidine at 0.2% ( $T_4$ ) and control ( $T_5$ ).

The insecticides were applied at fortnightly intervals from the pre-blooming stage. Hoppers population were counted and analysis was done after suit-

able transformation (2). Residues of endosulfan was studied in mango fruits after third (final) spray based on following procedure.

**Collection of Samples and Processing.** Fruit samples were collected on 0, 1, 5, 10, 15, 20 and 55 days after third application from all the plants, covering maximum fruits in the orchard. After collection fruit samples were chopped, mixed well and representative sample of 50 g was taken by quartering.

**Extraction and Clean Up Samples for Endosulfan Residues.** A representative 50 g of chopped fruit was taken for analysis. Then 100 ml of acetone was poured on the sample in 500 ml conical flasks and kept over night. The fruit sample was blended thoroughly in a Remi-automix blender for 2 minutes and the contents were filtered through Buchner funnel and rinsed with acetone repeatedly.

The extracts in acetone obtained from fruit samples were concentrated separately in rotary vacuum evaporator. The concentrated extract was subjected to solvent partitioning with n-hexane for three times (100 + 50 + 50 ml) after addition of 150 ml of distilled water and 10 g of NaCl. The n-hexane fractions were collected over anhydrous sodium sulfate and concentrated to about 5 ml using rotary vacuum evaporator.

**Table 1.** Effect of different treatments on hopper population and yield of mango.

Treatments	Population / 10 panicle at days after treatment						Overall mean population/10 panicle	Fruit/100 panicles at marble stage	Yield kg/plants at mature stage	Avoid-able yield loss (%)
	First spray		Second spray		Third spray					
	7	14	7	14	7	14				
No spray	96.80 (9.82)	108.80 (10.42)	119.00 (10.90)	173.00 (13.15)	291.20 (17.06)	233.00 (15.25)	170.30	40.20 (15.25)	9.5 (6.34)	
Carbaryl 0.15%	31.58 (5.60)	45.50 (6.73)	31.34 (5.59)	39.50 (6.27)	50.90 (7.12)	41.00 (6.38)	39.97	80.50 (8.97)	35.5 (5.95)	73.23
300 ppm Nimbicidine (0.2%)	33.98 (5.81)	50.60 (7.10)	37.70 (6.12)	46.28 (6.79)	56.84 (7.53)	45.5 (6.73)	45.15	83.28 (9.12)	29.8 (5.45)	68.12
Endosulfan (0.07%)	26.3 (5.10)	33.5 (5.77)	20.6 (4.52)	33.98 (5.82)	15.08 (3.86)	13.70 (3.67)	23.86	115.8 (10.76)	52.9 (7.27)	82.04
Endosulfan (0.14%)	23.3 (4.81)	28.28 (5.30)	16.70 (4.06)	25.10 (5.00)	29.96 (5.46)	20.9 (4.56)	24.04	130.9 (11.44)	58.4 (7.64)	83.73
SE ±	0.19	0.09	0.06	0.11	0.10	0.19		0.21	0.16	–
CD at 5%	0.87	0.41	0.27	0.50	0.46	0.87		0.96	0.73	–

The concentrated n-hexane fraction was quantitatively transferred on a glass column packed with a mixture of 10 g of silica gel and 1 g of activated charcoal. The collected solvent was then condensed in a rotary vacuum evaporator and transferred to a graduated tube and the volume was made up with distilled hexane for GC analysis.

**Estimation of Endosulfan Residues.** Gas Chromatograph, Hewlett Packard (USA) model 5890A coupled with an electron capture detector (ECD<sup>63</sup>Ni) and Chemito 5000 integrator were used for the analysis of endosulfan residue in different substrates. A megabore column, DB-1701 (length : 30 m, film thickness 1.5 µm, ID : 0.53 mm) was used separation of the endosulfan isomers and metabolites using nitrogen as the carrier gas with a flow rate of 30ml/minute. The oven, injector and the detector temperatures were maintained at 220C, 275C and 340C respectively.

An aliquot (1µl) of cleaned up extracts were injected into gas chromatograph with 10µl Hamilton syringe. The residues of endosulfan were identified by comparing the retention time of the sample peaks with those of a mixed standard solution containing 0.1 ppm each of α-endosulfan, β-endosulfan and endosulfan sulfate. The amount of residues present in different samples was then calculated by comparing the area of the peak of the respective component using the following formula :

Amount of residue in sample (µg/g) =

$$\frac{\text{Area of sample}}{\text{Area of standard}} \times \frac{\text{Amount of standard (ng) injected}}{\text{Weight of sample taken (g)}} \times \frac{\text{Total volume of sample (ml)}}{\text{Injected volume of sample}} \times \text{Recovery factor}$$

**Recovery Test.** Recovery study was carried out to establish the reliability of the analytical method and to assess the efficacy of extraction, partitioning and clean up steps by fortifying the substrates i. e. fruit with analytical grade endosulfan at the level of 0.2, 0.4 and 1 ppm. After necessary working up following the method as described under extraction and clean up of sample. The amount of endosulfan recovered from different substrates were analyzed by gas chromatography.

## Results and Discussion

Effect of different treatments on hopper population and yield of mango is presented in Table 1. Residue data for endosulfan are presented in Table 2. Considering the efficacy, endosulfan at both the treatment doses was found to be superior in controlling hopper population and yield increase over other treatments. Mean hopper population per 10 panicles was lowest (23.86) on plants treated with 0.07% endosulfan which was closely followed by 0.14% endosulfan treatment (24.04). Highest yield (130.9/10 panicles at

**Table 2.** Residue data for endosulfan 50 WPG in / on mango fruits.

Dose	Total residues of endosulfan ( $\alpha + \beta$ +sulphate) in ppm $\pm$ SD (average of 3 replication)							
	Days after application							
	0	1	5	10	15	20	55	60
0.07% a.i. (T <sub>1</sub> )	1.012 $\pm$ 0.14	0.243 $\pm$ 0.09	0.071 $\pm$ 0.02	0.046 $\pm$ 0.01	0.045 $\pm$ 0.01	0.027 $\pm$ 0.01	0.024 $\pm$ 0.01	ND
0.14% a.i. (T <sub>2</sub> )	1.253 $\pm$ 0.15	0.531 $\pm$ 0.05	0.362 $\pm$ 0.06	0.119 $\pm$ 0.01	0.058 $\pm$ 0.01	0.042 $\pm$ 0.01	0.038 $\pm$ 0.01	ND

**Table 2.** Continued.

Dose	Regression equation	t $\frac{1}{2}$ (days)	T <sub>MRL</sub> (days)
0.07% a. i. (T <sub>1</sub> )	Y = 2.2228 — 0.0207 X	2.73	13.8
0.14 % a. i. (T <sub>2</sub> )	Y = 2.5625 — 0.0237 X	2.95	20.02

marble stage and 58.4 kg/plant) was recorded in 0.14% of endosulfan treatment followed by 0.07% endosulfan dose (115.8/10 panicle at marble stage and 52.9 kg/plant). Similar trend was found in avoidable yield loss. Other treatments were also effective over control in all respects.

Regarding residue, initial deposit of total endosulfan was found to be 1.01 and 1.25 ppm for single and double dose respectively. The half-life value ( $t_{1/2}$ ) as calculated from regression equation was found to

be 2.73 days for single dose and 2.95 days for double dose. Furthermore, the safe waiting periods ( $T_{MRL}$ ) determined for endosulfan was 14—20 days.

#### References

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