

Persistence of Ethion Residues on Cucumber, *Cucumis sativus* (Linn.) using Gas Chromatography with Nitrogen Phosphorus Detector

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Received: 13 April 2007 / Accepted: 18 July 2007 / Published online: 16 August 2007
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Abstract Residues of ethion were estimated in cucumber by gas liquid chromatography following three applications of the insecticide at 375 and 750 g a.i ha⁻¹. The average initial deposits of ethion on cucumber fruits were found to be 2.40 and 4.97 mg kg⁻¹ at single and double dosages, respectively. Residues of ethion dissipated below the maximum residue limit (MRL) of 0.5 mg kg⁻¹ in 7 days. Half-life (T_{1/2}) for degradation of ethion on cucumber was observed to be 2.92 days at recommended dosage. A waiting period of 7 days is suggested for safe consumption of cucumber.

Keywords Cucumber · Ethion · MRL · Residues

Cucumber, [*Cucumis sativus* (Linn.)] is one of the important cucurbitaceous vegetable crops of North India. It is widely cultivated throughout India and other tropical and subtropical parts of the world. Cucumber is a rich source of vitamin B and C and consumed raw as a *salad* along with food. The low production of cucumber in the country is attributed to several factors, the most important being the damage caused by insect pests. The major pests responsible for losses in cucumber yield are spider mite, *Tetranychus cucurbitae* (Boisd), aphid, *Aphis gossypii* (Glover), red pumpkin beetle, *Raphidopalpa foveicollis* (Lucas), jassid, *Amrasca* sp. and fruit fly, *Bactrocera cucurbitae* (Coquillett). A number of organophosphate pesticides have been recommended against various pests of cucumber

(Anonymous 2005). Recently, ethion at 375 g a.i. ha⁻¹ has been found very effective against spider mite (Anonymous 2004). It is well recognized that there are risks attached to the consumption of pesticide-treated crops because of the presence of residues on them. Therefore, the rational recommendation of a pesticide requires that it must not only provide an effective control of pests but at the same time its residues on the commodity must also be toxicologically acceptable. At present there is no information available on the residues of ethion on cucumber under Indian conditions. There is an urgent need that the consumers be given information regarding the pesticide residues likely to be present in the food. Therefore, the present studies were undertaken to estimate the residues of ethion on cucumber following its applications at the minimum effective and double the minimum effective dosages.

Materials and Methods

Cucumber crop (var. Poinseete) was raised at the Entomological Research Farm, Punjab Agricultural University, Ludhiana, according to the recommended agronomic practices (Anonymous 2005). Nursery of cucumber was sown in the first week of February 2006. The seedlings were transplanted on ridges in plots of 100 m² in the third week of February 2006. There were three replications for each treatment including control arranged randomly. Control plots were sprayed with water only.

Ethion (Ethion 50 EC) was applied at minimum effective and double the minimum effective dosages of 375 and 750 g a.i. ha⁻¹, respectively. The pesticide was applied as high volume spray using Aspee Hand Knapsack Sprayer (capacity 15 L). The first application was made at 50% flowering followed by second and third applications at

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10 days interval. Spray volume used ranged from 75 to 150 L ha⁻¹ depending upon the stage of the crop.

Samples of cucumber were randomly taken from each treatment plot before and 0 (1 h after spraying), 1, 3, 5, 7, 10 and 15 days after the third application of insecticide. About five to six marketable size fruits of cucumber were collected at random from each plot. The samples were collected in polyethylene bags, labeled and brought to the laboratory for analysis. Each fruit sample was chopped, thoroughly mixed and reduced to appropriate sample size by successive sub-samplings. A gas liquid chromatographic method as described by Jyot et al. (2005) for extraction, clean up and final estimation of ethion on brinjal was followed with slight modifications. A representative sample of 200 g of cucumber was macerated in a blender and 50 g of this macerated sample was dipped in about 150 mL acetone and kept overnight. The extracted sample was filtered through glass wool plugged in a filtering funnel to 1 L separatory funnel alongwith rinsing of acetone. Diluted the filtrate with 600 mL of 2% aqueous solution of sodium chloride. Added 75 mL of dichloromethane to the separatory funnel and shaken thoroughly. The contents were allowed to stand till a clear separation of two phases was obtained. The lower dichloromethane layer was drained into a 500 mL beaker through anhydrous sodium sulfate supported on glass wool in a filtering funnel. The aqueous layer was re-extracted twice with 75 mL of dichloromethane each time and the lower organic phase was passed through the same anhydrous sodium sulfate and combined with the contents already obtained. The combined extracts thus obtained were treated with about 500 mg activated charcoal at room temperature for about 2–3 h. The clean extract thus obtained was filtered through Whatman filter paper No. 1 along with rinsings of acetone and was concentrated to about 4–5 mL on rotary vacuum evaporator at about 30°C.

The estimation of ethion residues was carried out by gas liquid chromatography equipped with NPD and a Pyrex glass column (1 m × 2 mm i.d.) packed with 3% OV-101 on Chromosorb W HP. The injector, detector and oven temperatures were maintained at 260, 270 and 230°C, respectively. The flow rates of nitrogen, hydrogen and zero air were set at 40, 20 and 100 mL min⁻¹, respectively. Under these operating conditions, ethion gave a distinct and sharp peak at retention time of 2.55 min. The limit of detection was found to be 0.01 mg kg⁻¹. Recovery experiments were carried out by spiking cucumber at different levels to establish the reliability and validity of analytical method adopted.

Cucumber fruits from control plots were spiked with ethion at levels of 0.25, 0.50 and 1.0 mg kg⁻¹ in triplicate. These were extracted, cleaned up and analyzed by following the method as described. The mean recoveries of

Table 1 Percent recovery of ethion from fortified samples of cucumber

| Level of fortification (mg kg ⁻¹) | Recovery, % ^a (mean ± SD) |
|---|--------------------------------------|
| 0.25 | 82.67 ± 2.31 |
| 0.50 | 86.67 ± 4.16 |
| 1.00 | 91.67 ± 1.53 |

^a Each value is the mean ± standard deviation of three replicate determinations

Table 2 Residues of ethion on cucumber following its application at 375 g a.i. ha⁻¹

| Days after treatment | Residue level (mg kg ⁻¹) | | Percent dissipation |
|----------------------|--------------------------------------|-------------|---------------------|
| | Replicates | Mean ± SD | |
| Before spray | BDL | | |
| | BDL | BDL | – |
| | BDL | | |
| 0 (1 h after spray) | 2.33 | | |
| | 2.41 | 2.40 ± 0.06 | – |
| | 2.46 | | |
| 1 | 1.63 | | |
| | 1.66 | 1.63 ± 0.03 | 32 |
| | 1.60 | | |
| 3 | 1.25 | | |
| | 1.20 | 1.24 ± 0.03 | 48.3 |
| | 1.27 | | |
| 5 | 0.72 | | |
| | 0.91 | 0.84 ± 0.10 | 65 |
| | 0.90 | | |
| 7 | 0.42 | | |
| | 0.38 | 0.40 ± 0.02 | 83.3 |
| | 0.40 | | |
| 10 | 0.23 | | |
| | 0.25 | 0.24 ± 0.01 | 90 |
| | 0.24 | | |
| 15 | BDL | | |
| | BDL | BDL | – |
| | BDL | | |

BDL Below detectable limit (<0.01 mg kg⁻¹)

ethion fortified at these levels were found to be consistent and more than 80% (Table 1).

Results and Discussion

The mean initial deposits of ethion on cucumber fruits were found to be 2.40 and 4.97 mg kg⁻¹ following three applications at 10 days interval at 375 and 750 g a.i. ha⁻¹, respectively. More than 80% and 90% of these ethion

Table 3 Residues of ethion on cucumber following its application at 750 g a.i. ha⁻¹

| Days after treatment | Residue level (mg kg ⁻¹) | | Per cent dissipation |
|----------------------|--------------------------------------|-----------------|----------------------|
| | Replicates | Mean \pm SD | |
| Before spray | BDL | | |
| | BDL | BDL | – |
| | BDL | | |
| 0 (1 h after spray) | 4.90 | | |
| | 5.04 | 4.97 \pm 0.07 | – |
| | 4.98 | | |
| 1 | 2.97 | | |
| | 2.99 | 2.98 \pm 0.01 | 40 |
| | 2.98 | | |
| 3 | 2.38 | | |
| | 2.36 | 2.35 \pm 0.03 | 52.7 |
| | 2.31 | | |
| 5 | 1.60 | | |
| | 1.68 | 1.64 \pm 0.04 | 67 |
| | 1.64 | | |
| 7 | 0.86 | | |
| | 0.88 | 0.89 \pm 0.03 | 82 |
| | 0.93 | | |
| 10 | 0.28 | | |
| | 0.44 | 0.39 \pm 0.09 | 92.1 |
| | 0.45 | | |
| 15 | BDL | | |
| | BDL | BDL | – |
| | BDL | | |

BDL Below detectable limit (<0.01 mg kg⁻¹)

residues dissipated after 7 and 10 days, respectively, at single and double dosages (Tables 2, 3). Half-life ($T_{1/2}$) values for degradation of ethion on cucumber fruits were calculated as per Hoskin 1961 and observed to be 2.92 and 3.04 days, respectively, at recommended and double the recommended dosages. Maximum residue limit (MRL) of ethion on cucumber has been prescribed as 0.50 mg kg⁻¹ (Handa et al. 1999). Residues of ethion on cucumber fruits were less than its MRL value after 7 days of its application at the recommended dosage.

MacNeil and Hikichi (1976) studied residues of ethion on pear fruits following its application (8 lb 25 WP ethion) for the control of pear psylla. Ethion residues remained within its MRL of 1 mg kg⁻¹ after 20–30 days of its application.

Residues of ethion were estimated in onion after in-furrow treatment at different rates, i.e. 2.2, 2.3 and

4.5 kg ha⁻¹. It was observed that ethion residues exceeded their MRL value of 0.1 mg kg⁻¹ in all the treatments when analyzed 86 days after its treatment at harvest time (Braun et al. 1980).

Arora et al. (2004) also estimated residues of ethion on pear fruits following its application at 0.05% and 0.1% a.i. Average initial deposits were observed to be 1.53 and 3.11 mg kg⁻¹, respectively. These residues were less than the MRL value of 2.0 mg kg⁻¹ of ethion on pear fruits. Half-life of ethion was observed to be 1.95 and 2.21 days at single and double the dosages, respectively.

Ethion was applied at 375 and 750 g a.i. ha⁻¹ on brinjal and average initial deposits were found to be 2.01 and 4.93 mg kg⁻¹, respectively. Residues of ethion dissipated below its MRL value of 1.0 mg kg⁻¹, after 4 days of its application at recommended dosage (Jyot et al. 2005).

Therefore, the results of the present study indicate that residues of ethion degrade below its MRL of 0.50 mg kg⁻¹, after 7 days of its application at the recommended dosage. So it is suggested that a waiting period of 7 days should be observed before consumption of cucumber fruits, as it will be safe for the consumers health.

Acknowledgments Authors thank the Professor and Head, Department of Entomology P.A.U Ludhiana for providing the necessary research facilities during the tenure of study.

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