

## Maximum Residue Limit and Risk Assessment of Beta-Cyfluthrin and Imidacloprid on Tomato (*Lycopersicon esculentum* Mill)

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Received: 31 October 2002/Accepted: 9 March 2003

Crop protecting chemicals have been performance maximizer in achieving better farm productivity. These have been the most powerful tool available to mankind for combating pests of agriculture and public health. Simplicity, efficiency and economic return have been the hall mark of their wide use. As a result of continuous use, at times their residues find the place in edible parts, soil, water and other environment.

In order to protect the health of the consumer while facilitating international trade, the primary objective is to develop the Maximum Residue Limit (MRL). Public health considerations are taken into account by establishing the MRLs at level not higher than those resulting from the use of pesticide in accordance with Good Agricultural Practices (GAP). However, explicit consideration of possible exposure to residues of a pesticide is an integral part of the risk assessment process to ensure that the Acceptable Daily Intake (ADI) of the pesticide is not exceeded. The best assurance that exposure to residues are within safe limits is obtained from dietary intake studies but when such studies are not feasible or the pesticide is not being used or has only recently been approved for use, pesticide residues intake must be predicted on the basis of available data (Anonymous 1989; 1997).

In India, imidacloprid is registered for use in plant protection practices and the use of beta-cyfluthrin, a recently introduced fluorinated pyrethroid, is presently approved/registered for cotton crop. The MRLs for these insecticides on tomato have not been fixed in our country. Therefore, an attempt has been made to fix their MRL on tomato and to study risk assessment by generating food chemical concentration (or residues) data through supervised field trials conducted for successively three crop years.

### MATERIALS AND METHODS

Tomato (*Lycopersicon esculentum* Mill) crop (variety Pusa Sheetal) was transplanted on raised beds (4.5 x 0.5 m<sup>2</sup>) during 1999-2001 at the experimental farm of Indian Agricultural Research Institute, New Delhi (India). Plant spacing was 45 x 45 cm<sup>2</sup>. Beta-cyfluthrin is recommended especially for Lepidopteran

pests like *Helicoverpa armigera* and also against Coleoptera, Hemiptera and Homoptera (Anonymous 1992; Juliedes et al. 1992; Pap et al. 1996 and Tomlin 2000) at variable dosages from 7.5 to 25.0 g ai ha<sup>-1</sup>. Similarly imidacloprid is reportedly effective against sucking pests particularly aphid, *Aphis gossypii* and leaf hopper *Amrasca biguttula biguttula* and white fly, *Bemisia tabaci* (Elbert et al. 1990; Kumar 1999; Anonymous 2000; Tomlin 2000). This has been found effective at varying doses of 15 -100 g ai ha<sup>-1</sup> as foliar spray. In view of this, beta-cyfluthrin (Bulldock 025 SC, Bayer India Limited, New Delhi, India) was sprayed on tomato crop @ 18.75 (recommended dose) and 37.50 g ai ha<sup>-1</sup> (double the recommended dose) during the trial of 1999 and @ 12.50, 18.75 and 37.50 g ai ha<sup>-1</sup> for 2000. During third year trial (2001), the dosages were same as of 2000 but an additional dose of 75 g ai ha<sup>-1</sup> was also taken to see the adverse effect and plant compatibility.

In a separate experiment, imidacloprid (diluted from Confidor 200SL, Bayer India Limited, New Delhi, India) was sprayed @ 20 and 40 g ai ha<sup>-1</sup> for three year trials. However, imidacloprid @ 80 g ai ha<sup>-1</sup> was also applied during 2001 to see phytotoxicity/plant compatibility. The insecticides beta-cyfluthrin and imidacloprid were sprayed on the tomato crop when some fruits had set in. Fifteen days later, a second spray was given. The fluid rate was 500 L ha<sup>-1</sup> for both the sprays/insecticides.

For determining beta-cyfluthrin and imidacloprid concentration in tomato fruits at different intervals, the fruit samples (200 g) were collected randomly from each treatment at 0 (1 hr after insecticidal spray), 1, 3, 5, 7, 10, 15, 20, 25 and 30 days after second spray only from all the trials. The fruit samples were cut into small pieces (approx. 1x1 cm<sup>2</sup>), mixed and a sub sample of 25g was taken for further processing/analysis. Soil samples (0-10 and 10-20 cm deep cores, 500g) were collected after fruiting season to examine the movement/leaching in the soil. Soil samples were air dried, ground and sieved and a sub sample of 25 g was taken for analysis.

The method for extraction, clean up and analysis of fruit and soil samples described by Kumar (1999); Kumar and Dikshit (2001); Dikshit et al. (2002) was followed for determining the residues of imidacloprid by HPLC, while for beta-cyfluthrin the method of Blass 1987; Dikshit et al. (2001a and 2002) was utilized for quantification of the residues through GLC. The recoveries of beta-cyfluthrin and imidacloprid from spiked tomato fruits were 82 to 88 and 80 to 84 percent, respectively; 83 to 85 and 87 to 89 percent from soil.

Insecticides on raw commodities are normally dissipated during storage, transport, preparation, commercial processing or cooking/steaming. Some pesticides are destroyed by food preparation processes (Anonymous 1997; Dikshit 1985; Dikshit et al. 2001 a, b) and many primary products are consumed only after processing. Generally tomato fruits are washed with water and then either consumed as raw in salad dressing or in ketchup, puree, tastemaker in recipes, soup and in other forms. Therefore, effect of washing the fruits with water

followed by cooking/steaming was seen to lead to a reduction in the residues of beta-cyfluthrin and imidacloprid. This effect was seen only from 0 and 3 day contaminated samples from second year trial (2000).

Since the MRL of beta-cyfluthrin and imidacloprid are not available on tomato in India, an effort was made to establish safety factors like Pre-harvest Intervals (PHI), Theoretical Maximum Residue Contribution (TMRC), National Theoretical Maximum Daily Intake (NTMDI), Predicting Dietary Intake (PDI) and Processing factor (PF) in order to arrive at risk assessment to human health from analytical data.

## RESULTS AND DISCUSSION

The data on the occurrence of beta-cyfluthrin availability in/on tomato fruits at varied time are presented in Table 1.

**Table 1.** Availability of beta-cyfluthrin in/on tomato from foliar spray.

| Days        | Treatment<br>(g ai ha <sup>-1</sup> ) | Amount of beta - cyfluthrin (mg kg <sup>-1</sup> ) |              |              |
|-------------|---------------------------------------|--|--------------|--------------|
|             |                                       | 1999   | 2000         | 2001         |
| 0<br>(1 hr) | 12.50                                 | -  | 0.42         | 0.58         |
|             | 18.75                                 | 1.12   | 0.95         | 1.00         |
|             | 37.50                                 | 2.10   | 2.35         | 1.90         |
| 1           | 12.50                                 | -  | 0.34 (19.04) | 0.40 (32.14) |
|             | 18.75                                 | -  | 0.76 (20.00) | 0.64 (36.00) |
|             | 37.50                                 | -  | 1.85 (21.27) | 1.00 (47.36) |
| 3           | 12.50                                 | -  | 0.26 (38.09) | 0.23 (60.34) |
|             | 18.75                                 | 0.60 (46.42)                                       | 0.44 (53.68) | 0.38 (62.00) |
|             | 37.50                                 | 1.28 (39.04)                                       | 1.50 (36.17) | 0.62 (67.36) |
| 5           | 12.50                                 | -  | 0.10 (76.19) | 0.08 (86.20) |
|             | 18.75                                 | 0.30 (73.21)                                       | 0.24 (74.73) | 0.18 (82.00) |
|             | 37.50                                 | 0.68 (67.61)                                       | 0.72 (69.36) | 0.40 (78.94) |
| 7           | 12.50                                 | -  | 0.04 (90.47) | ND (100.00)  |
|             | 18.75                                 | 0.08 (92.85)                                       | 0.10 (89.47) | 0.05 (95.00) |
|             | 37.50                                 | 0.30 (85.71)                                       | 0.22 (90.63) | 0.14 (92.63) |
| 10          | 12.50                                 | -  | ND (100.00)  | ND (100.00)  |
|             | 18.75                                 | ND (100.00)  | ND (100.00)  | ND (100.00)  |
|             | 37.50                                 | 0.04 (98.09)                                       | 0.05 (97.87) | ND (100.00)  |
| 15          | 12.50                                 | -  | ND (100.00)  | ND (100.00)  |
|             | 18.75                                 | ND (100.00)  | ND (100.00)  | ND (100.00)  |
|             | 37.50                                 | ND (100.00)  | ND (100.00)  | ND (100.00)  |

ND, Non detectable; Figures in parentheses are percent cumulative loss.

The initial deposit of beta-cyfluthrin on tomato fruits was 0.42-0.58 mg kg<sup>-1</sup> and 0.95-1.12 mg kg<sup>-1</sup> from 12.50 and 18.75 g ai ha<sup>-1</sup> treatments in all the trials. However, the initial concentration was 1.90-2.35 mg kg<sup>-1</sup> in tomato fruits from higher rate of application (37.50 g ai ha<sup>-1</sup>). The reported concentration declined

progressively with time from all the treatments/trials and became non detectable after 7 days of application. The initial concentration obtained on fruits were low due to major fall of spray quantity on plant foliage, attachment of fruits under dense foliage and phenotypic character of fruits. The ADI (Acceptable Daily Intake) of beta-cyfluthrin is 0.02 mg kg<sup>-1</sup> body weight, toxicity class, WHO (a.i.) 1b; EPA (formulation) II. Therefore MPI value (Maximum Permissible Intake) i.e. maximum permissible quantity of the insecticide for a human being (50 kg, intentional low weight consideration) per day during a part or whole life is 1 mg person<sup>-1</sup> day<sup>-1</sup>; for a person of 55 kg weight, the MPI is 1.1 mg person<sup>-1</sup> day<sup>-1</sup>. The data on the recommended tomato consumption in balanced Indian diet is not available, but total vegetable consumption (other than roots, tubers and green leafy vegetables) is about 100g (Anonymous 1999; Subbian et al. 2000). Since tomato is consumed in various forms as mentioned earlier and assuming 100g intake of tomato in lieu of total vegetables, the TMRC/TMDI value from fruit samples collected on 0 day (after 1 hr of treatment) from recommended dose i.e. 18.75 g ai ha<sup>-1</sup> (based on bioefficacy data generated in India) found to be 0.101 mg person<sup>-1</sup> day<sup>-1</sup> (based on three year trials supervised median pesticide residues). The calculated TMDI value is much lower than the MPI, therefore beta-cyfluthrin treatment of recommended dose appears safe and consumption of such tomatoes are not likely to risk the health. Even if higher treatment rate (37.50 g ai ha<sup>-1</sup>) and the corresponding median pesticide residue value is considered the TMDI value is found lower than the MPI value, therefore higher dose, if used inadvertently would also not probably cause harm to consumers. But still to have more margin of safety (MOS) and as a normal convention in India, the tomato fruits are not plucked just after the spray of insecticide. The fruits are normally harvested at an interval of 3 days and minimum 3 days after spray. In view of this, the TMRC /TMDI values from the recommended treatment (18.75 g ai ha<sup>-1</sup>) based on STMPR (Supervised trial median pesticide residues) from 3 year data, on third day, found to be 0.046 mg person<sup>-1</sup> day<sup>-1</sup>. This value is still much lower than the MPI value and also lower than TMRC/TMDI from fruits after 1 hour of spray. Further, the highest possible tolerance or MRL (toxicologically) was found to be about 4 ppm (based on total diet 1.5 kg and treated tomato 100g) but median pesticide residues derived from supervised trials (from three years) from GAP after 3 days of spray (from recommended dose, 18.75 g ai ha<sup>-1</sup>) were 0.46 mg kg<sup>-1</sup>. This value is much lower than the highest possible tolerance (toxicologically). Therefore, beta-cyfluthrin can be assigned MRL of 0.5 mg kg<sup>-1</sup> on tomato fruits and PHI of 3 days is suggested after the spray of beta-cyfluthrin. Assuming the MRL of beta-cyfluthrin in India on tomato is fixed as 0.5 mg kg<sup>-1</sup> and consumption of tomato as 100g (total vegetable basis other than roots, tubers and green leafy vegetables), the NTMDI value is found 0.05 mg person<sup>-1</sup> day<sup>-1</sup> which is equivalent to 5% of total ADI. Hence, beta-cyfluthrin treatment at recommended dose is found very safe.

The PF from zero day (1 hr after spray) contaminated samples when washed with water was recorded as 0.57-0.59 (rounded to 0.6). This indicated further safety but not to the extent of safe level if proposed MRL of 0.5 mg kg<sup>-1</sup> is considered. However, the PF of 0.41-0.43 (rounded to 0.4) was recorded from 0 day samples

when washed and steamed. Therefore, 0 day samples, if washed and steamed, rendered the residues below the proposed MRL from the recommended dose and hence not likely to risk the health. Besides, the PF factors obtained from 3 day contaminated samples, if considered, it will enhance two fold MOS.

The initial concentration of imidacloprid detected in tomato fruits declined with time from all the treatments/trials. Similar rate of dissipation between the two doses was not dependent on the dose of imidacloprid and followed first order rate kinetics. However, a close scrutiny of the data (Table 2) reveals that for recommended dose 20 g ai ha<sup>-1</sup>, 50-55% insecticide was lost within 3 days of application and the remaining quantity took another 7 days to obtain 95-100% dissipation. Similarly for higher rate of application 54-68% imidacloprid was lost in 3 days time and the remaining got dislodged in a further period of 7-12 days. This shows that imidacloprid followed a biphasic dissipation i.e. at a faster rate during initial days followed by slow and steady pace after application on tomato.

**Table 2.** Amount of imidacloprid in/on tomato from foliar spray.

| Days   | Treatment<br>(g ai ha <sup>-1</sup> ) | Amount of imidacloprid (mg kg <sup>-1</sup> ) |              |              |
|--------|---------------------------------------|---|--------------|--------------|
|        |                                       | 1999  | 2000         | 2001         |
| 0      | 20                                    | 1.35  | 1.00         | 0.75         |
| (1 hr) | 40                                    | 2.40  | 2.12         | 1.80         |
| 1      | 20                                    | -   | 0.60 (40.00) | 0.60 (20.00) |
|        | 40                                    | -   | 1.45 (31.60) | 1.00 (44.44) |
| 3      | 20                                    | 0.60 (55.55)                                  | 0.45 (55.00) | 0.38 (49.33) |
|        | 40                                    | 1.10 (54.16)                                  | 0.90 (57.54) | 0.56 (68.88) |
| 5      | 20                                    | 0.22 (83.70)                                  | 0.28 (76.00) | 0.18 (76.00) |
|        | 40                                    | 0.50 (79.16)                                  | 0.68 (70.75) | 0.32 (82.22) |
| 7      | 20                                    | 0.08 (94.07)                                  | 0.14 (90.00) | 0.07 (90.66) |
|        | 40                                    | 0.18 (92.50)                                  | 0.26 (82.67) | 0.16 (91.11) |
| 10     | 20                                    | ND (100.00)                                   | 0.05 (95.00) | ND (100.00)  |
|        | 40                                    | 0.06 (97.50)                                  | 0.08 (96.22) | ND (100.00)  |
| 15     | 20                                    | ND (100.00)                                   | ND (100.00)  | ND (100.00)  |
|        | 40                                    | ND (100.00)                                   | ND (100.00)  | ND (100.00)  |

ND, Non detectable; Figures in parentheses are percent cumulative loss.

During the analysis it was found that though the quantities of both the insecticides in tomato were in resembling range but beta-cyfluthrin was mostly available on the surface or in the epicarp of the fruits while imidacloprid was detected after 24 hr in the mesocarp and endocarp also from all the days of sampling. This indicated that beta-cyfluthrin probably does not get translocated inside the fruits as it has no systemic action while a reverse was found true for imidacloprid.

The ADI of imidacloprid is 0.057 mg kg<sup>-1</sup> body weight. The MPI is calculated to be 2.85 mg kg<sup>-1</sup>. Based on the same analogy and approach (explanation) as described for beta-cyfluthrin, the TMRC/TMDI of imidacloprid was recorded 0.10 and 0.22 mg person<sup>-1</sup> day<sup>-1</sup> from 0 day contaminated samples from both the

treatments based on STMPR values of three years data. These values are less than MPI, therefore application of imidacloprid appears safe. Since the crops are sprayed to saturation and drenched (Machado-Neto et al. 1992), and as a convention fruits are not plucked for consumption just after spray in order to increase MOS. Hence, deriving TMRC/TMDI from 3<sup>rd</sup> day samples would be very practical and accordingly from the recommended dose, it was found to be 0.042 mg person<sup>-1</sup> day<sup>-1</sup> based on STMPR which is much less than MPI. Therefore MRL of 0.5 mg kg<sup>-1</sup> can be assigned for imidacloprid on tomato based on STMPR of 0.42 mg kg<sup>-1</sup> (rounded significant figure, 0.5) obtained from recommended dose and GAP. PHI of 3 days is suggested after spray of application of imidacloprid. Taking into account the MRL of 0.5 mg kg<sup>-1</sup> for imidacloprid on tomato, NTMDI is recorded as 0.05 which corresponds 1.75% of total ADI (rounded to significant higher value 2%). Therefore imidacloprid treatments are found safe in plant protection practices.

In the case of imidacloprid the PF was found 0.60-0.61 (rounded to 0.6) from washing of samples with water while 0.39-0.40 (rounded to 0.4) from washing followed by steaming. Washing alone did not bring the residues below assigned MRL (0.5 mg kg<sup>-1</sup>), however washing + steaming dislodged the residues to safe level from recommended application. If PF from 3 days contaminated samples is considered the consumption will lead to extreme MOS.

The residues of both the insecticides were non detectable in soil samples (0-10, 10-20 cm) collected after fruiting season of the crop.

The half-life of beta cyfluthrin ranged from 1.21 to 2.18 days, while imidacloprid dissipated with the half-life of 1.71 to 2.43 days from all the treatments/trials (Table 3).

The 4 X doses of 75 g ai ha<sup>-1</sup> of beta-cyfluthrin and 80 g ai ha<sup>-1</sup> of imidacloprid did not exhibit any phytotoxic symptom and plant compatibility was good. No adverse effect on beneficial insects like *coccinella* was noticed.

**Table 3.** Half-life (days) of beta-cyfluthrin and imidacloprid on tomato.

| Insecticide                   |      | Beta-cyfluthrin |       |       | Imidacloprid |      |
|-------------------------------|------|-----------------|-------|-------|--------------|------|
| Dose (g ai ha <sup>-1</sup> ) |      | 12.50           | 18.75 | 37.50 | 20           | 40   |
| Year                          | 1999 | -               | 1.90  | 1.76  | 1.71         | 1.82 |
|                               | 2000 | 2.07            | 2.18  | 1.21  | 2.43         | 2.19 |
|                               | 2001 | 1.80            | 1.71  | 2.05  | 2.07         | 2.10 |

The bioeffectivity of the insecticides was adjudged by recording control of leaf hopper, *Amrasca bigutulla bigutulla* and fruit borer, *Helicoverpa armigera*. The imidacloprid treatments harboured less leaf hopper population and beta-cyfluthrin manifested significantly less fruit damage when compared with no treatment. About 70-74% leaf hopper control and 20-24% less damage due to fruit borer was achieved as compared to no treatment.

To conclude, both the insecticides were safe at the experimental dosages and can fit in the efficient plant protection practices without any ill traits on the environment. The doses of 18.75 g ai ha<sup>-1</sup> and 20 g ai ha<sup>-1</sup> of beta-cyfluthrin and imidacloprid, respectively were found optimum as foliar spray. PHI of 3 days and MRL of 0.5 mg kg<sup>-1</sup> for both the insecticides is recommended for tomato crop. PF showed almost double fold MOS.

*Acknowledgment.* We thank Dr. B. S. Parmar, Head, Division of Agricultural Chemicals for sustained interest in the studies.

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