Asian Australas. J. Food Saf. Secur. 2022, 6 (1), 27-34; https://doi.org/10.3329/aajfss.v6i1.59783

Asian-Australasian Journal of Food Safety and Security
ISSN 2523-1073 (Print) 2523-2983 (Online)
https://www.ebupress.com/journal/aajfss/

Article

Dissipation of dimethoate and fenitrothion in yard long bean and tomato under supervised field trials

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Received: 30 March 2022/Accepted: 25 May 2022/ Published: 30 May 2022

Abstract: The study was conducted to detect and quantify the residue of dimethoate and fenitrothion in yard long bean and tomato depending on the maximum residue limit (MRL) set by European Union. Four supervised field trials were conducted and sprayed with the recommended dose (2 ml/L of water) of dimethoate and fenitrothion. Samples were collected at 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 and 11days after spray (DAS). The collected samples were analyzed using Gas Chromatography (GC) coupled with Electron Capture Detector (ECD). The residue of fenitrothion was detected up to 8 DAS in tomato and 9 DAS in yard long bean and the levels of residues were 8.983-0.076 mg/kg in tomato and 6.187- 0.056 mg/kg in yard long bean which were above MRL. No residue was detected at 9 DAS in tomato and 10 DAS in yard long bean. The residue of dimethoate was detected up to 9 DAS in tomato and 10 DAS in yard long bean. The quantities of residues were decreased gradually over time and these were 6.521- 0.034 mg/kg in tomato and 8.590-0.046 mg/kg in yard long bean which were above MRL. No residues were detected at 10 DAS in tomato and 11 DAS in yard long bean.

Keywords: dissipation; dimethoate; fenitrothion; vegetables; MRL; gas chromatography

1. Introduction

The yard long bean (*Vigna unguiculata* L.) is a legume in the family Fabaceae, cultivated to be eaten as green pods. It is also known as borboti, chinese long bean, snake bean, asparagus bean, black-eye bean and long-podded cowpea (John and Lincoln, 2022). Customers prefer 10-12 inches in length pods before the seeds mature and expand. Although both red seeded and black seeded yard long bean exist, the black seeded pod is preferred for human consumption. The tomato (*Solanum lycopersicum*) is a plant in the family Solanaceae, cultivated extensively for its edible fruits. Labeled as vegetables for nutritional purposes, tomatoes are a good source of vitamin C and the phytochemical lycopene (Gloria Lotha, 2019). The fruits are normally eaten raw in salads, served as a cooked vegetable, used as an ingredient of various prepared dishes, and pickled.Both the vegetables are nutritious and commercial crop in Bangladesh. These crops are attacked by a many insect pests. Fenitrothion and dimethoate are an organophosphorus insecticide. These are one of the major components of plant protection for the farmers of Bangladesh because of its small quantity in packaging/bottling, storage ability and availability in the market. As crop production and insecticides are closely related and their left over residue might or might not persist in the environment that should be carefully examined and monitored. Most of the time insecticides are being used by vegetable farmers irrationally. It was understood from farmers' interview that they use insecticides indiscriminately (Anon. 2001; Ahmed *et al.* 2005). A considerable number of farmers sell vegetables immediate after spray or at an interval of 0-2 days after spray (Anon. 2000). This led to assume that over-sprayed vegetable consumers might face health hazards and environment might be over loaded with insecticide residue. Chemical pesticide can become hazard to the users, consumers and the environment. Pesticide can produce negative impacts, both social and private (Antle and Pingali, 1994). In Bangladesh, very little endeavor have been made to document pesticide residue load in our agro ecological condition. Monitoring of pesticide residues in vegetables and fruits is being done regularly in abroad (Krol *et al.* 2000; Van der Schee, 2002; Kumar *et al.* 2004; Rajeswaran *et al.* 2004) and on and off basis in Bangladesh (Khatoon *et. al.*, 2004; Kabir *et al.*, 2008; 2008a; Ahmed *et al.*, 2016a; 2016b; 2018a; 2019; 2021a; Ismail *et al.*, 2019; 2019a; 2019b; Aktar *et al.*, 2017; Habib *et al.* 2021; Hasan *et al.*, 2021; 2017; Islam *et al.*, 2021 ; Islam *et al.*, 2014; Nahar *et al.*, 2020; Parven *et al.*, 2021; Parvin *et al.*, 2021). Extensive use of pesticides has resulted in contamination of air, water and food. Pesticide residues in food have become a consumer's safety issue and the consumers have the right to know how much pesticides remain in the food. But scanty of references are available on the study of residue degradation of pesticides in vegetables in Bangladesh (Prodhan *et al.*, 2018; Ahmed *et al.*, 2018; 2021). Considering these, the present study was undertaken to assess the amount and dissipation rate of left over residues of two frequently used insecticides in yard long bean and tomato.

2. Materials and Methods

The standard for dimethoate and fenitrothion were obtained from Sigma-Aldrich Laborchemikalien, (Gmbh P O Box-100262 D-30918, Seelze, Germany) via Bangladesh Scientific Pvt. ltd. Dhaka, Bangladesh. Standards of both insecticides contained 99.6% purity. Marketable size of yard long bean and tomato were collected from four different supervised field trials at 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 and 11 days after spray (DAS) which were sprayed with fenitrothion and dimethoate at the rate of 2ml/L of water. The formulated products of those were Sumithion 50ECand Tafgor 40EC. The purity of formulated insecticides were tested in the pesticide analytical laboratory (ISO/IEC 17025: 2017 accredited) and found 100% pure. Methanol, acetone, gradient grade acetonitrile, sodium chloride (NaCl), anhydrous magnesium sulphate ($MgSO_4$) and Primary Secondary Amine (PSA) were also purchased from Bangladesh Scientific Pvt. ltd. Dhaka, Bangladesh.

2.1. Extraction, separation and clean up

The QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method was used to extract insecticides from vegetables. The method was slightly modified and adopted. Collected vegetable samples were chopped on a chopping board with a sharp knife and grounded using laboratory grinder. 10 g of chopped sample was transferred into a 50 ml centrifuge tube. Then 10 ml of acetonitrile (Sigma-Aldrich, Analytical grade) was added into centrifuge tube and shook well for proper mixing. Again 4 grams of anhydrous $MgSO_4$ (Scharlau, Analytical grade) and 1g of NaCl (Sigma-Aldrich, Analytical grade) were added and vigorous shaking was done for 1 minute. Then centrifuge tube containing sample with chemical mixture was kept at centrifuge machine (Sigma-3K30, Germany) and centrifuged at 5000 rpm for 5 minutes. After centrifugation 2 ml of the supernatant was transferred into an Eppendorf tube containing 100 mg PSA, 150 mg MgSO4 and 100 mg of charcoal for cleaning up and shook it vigorously for 2 minutes. Eppendorf tubes containing sample extract was placed in centrifuge machine and centrifuged at 5000 rpm for 5 minutes. After centrifuge, a 1 mL of sample extract was filtered through a 0.45 µm PTFE filter using a syringe and transferred into vial for analysis in Gas Chromatography (GC).

2.2. Detection and Quantification of pesticide residue in samples

The concentrated extracts were subjected to analysis by GC-2010 (Shimadzu). Flame Thermionic Detector (FTD) was used for the quantification of organophosphorus insecticide (fenitrothion and dimethoate) in GC. Separations were done by capillary column ATTM-1 (30 m long, 0.25 mm inner diameter and 0.25µm film thicknesses). Helium was used as carrier and make up gas. The instrument parameters for GC-FTD were as follows: Injection port temperature: 260^0C, purge flow: 3ml/min, split ratio: 20. Column oven initial temperature: 160^0C which went up to 240^0C following 8 min incremental time. Detector temperature: 280^0C, stop time: 12 min, current: 0.5pA, makeup flow: 30 ml/min, H_2 flow: 1.5 mL/min and air flow was 145 ml/min.
Prior to the injection of the sample extract, standard solutions of different concentrations of both pesticides were prepared and injected with the above instrument parameters. The samples were calibrated (retention time, peak area etc.) against three to four pointed calibration curve of standard solution of concerned pesticide. Each peak was characterized by its retention time. Sample results were expressed in mg/kg automatically by the GC

software which represented the concentration of the final volume injected. From this value the actual amount of pesticide residue present in the sample was determined by using the following formula.

$$\text{Residue in sample (mg/kg)} = \frac{\text{Conc. obtained in injected volume (mg/kg) X Quantity of final volume (L)}}{\text{Amount of sample taken (kg)}}$$

3. Results

3.1. Dimethoate

The yard long bean and tomato samples containing residues were analyzed using the GC-FTD with the developed parameters. The results obtained from this study have been summarized in Tables 1 and 2.

Table 1. Quantity of residue of dimethoate (Tafgor 40EC) estimated from yard long bean.

Days after spraying	Total volume prepared	Injected volume (µL)	Amount of Residue (mg/kg)	EU MRL (mg/kg)
0	10	1	8.590	0.01
1	10	1	7.021	
2	10	1	6.175	
3	10	1	4.350	
4	10	1	3.104	
5	10	1	2.053	
6	10	1	1.042	
7	10	1	0.769	
8	10	1	0.461	
9	10	1	0.074	
10	10	1	0.046	
11	10	1	ND	

The left over residue of dimethoate in the yard long bean sample had been detected up to 10 DAS. At 0 DAS, the quantity of residue was 8.590 mg/kg and they were 7.021 mg/kg, 6.175 mg/kg, 4.350 mg/kg, 3.104 mg/kg, 2.053 mg/kg, 1.042 mg/kg, 0.769 mg/kg and 0.461 mg/kg, 0.074 mg/kg and 0.046 mg/kg at 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 DAS, respectively. All these quantities were above MRL set by European Union. No residue was detected at 11 DAS. So, yard long bean can be harvested safely at 11 DAS.

Table 2. Quantity of residue of dimethoate (Tafgor 40EC) estimated from tomato.

Days after spraying	Total volume prepared	Injected volume	Amount of Residue (mg/kg)	EU MRL (mg/kg)
0	10	1	6.521	0.01
1	10	1	4.319	
2	10	1	2.986	
3	10	1	1.578	
4	10	1	0.986	
5	10	1	0.564	
6	10	1	0.309	
7	10	1	0.165	
8	10	1	0.062	
9	10	1	0.034	
10	10	1	ND	

The results revealed that residue of dimethoate in tomato sample had been detected up to 9 DAS. The quantities of residue were above MRL up to 9 DAS and these were 6.521 mg/kg, 4.319 mg/kg, 2.986 mg/kg, 1.578 mg/kg, 0.986 mg/kg, 0.564 mg/kg, 0.309 mg/kg, 0.165 mg/kg, 0.062mg/kg and 0.034 mg/kg at 0, 1, 2, 3, 4, 5, 6, 7, 8, and 9 DAS, respectively. All of the tested samples showed residues which were above MRL set by European Union. No residue was detected at 10 DAS. So, tomato can be harvested safely at 10 days after spray.

3.2. Fenitrothion

The yard long bean and tomato samples containing fenitrothion residue were analyzed using the GC-FTD with developed parameters. The results obtained from this analysis have been summarized in Tables 3 and 4.

Table 3. Quantity of residue of fenitrothion (Sumithion 50EC) estimated from yard long bean.

Days after spraying	Total volume prepared	Injected volume	Amount of Residue (mg/kg)	EU MRL (mg/kg)
0	10	1	6.187	0.01
1	10	1	4.218	
2	10	1	3.236	
3	10	1	2.098	
4	10	1	1.262	
5	10	1	0.565	
6	10	1	0.349	
7	10	1	0.106	
8	10	1	0.084	
9	10	1	0.056	
10	10	1	ND	

From the Table 3, it was observed that residue of fenitrothion could be detected up to 9 DAS. The quantities of residue were 6.187 mg/kg, 4.218 mg/kg, 3.236 mg/kg, 2.098 mg/kg, 1.262 mg/kg, 0.565 mg/kg, 0.349 mg/ kg and 0.106 mg/kg, 0.084 mg/kg and 0.056 mg/kg at 0, 1, 2, 3, 4, 5, 6, 7, 8 and 9 DAS, respectively. All of the samples had residues which were above MRL set by European Union. No residue was detected at 10 DAS. So, yard long bean can be harvested safely at 10 days after spray.

Table 4. Quantity of residue of fenitrothion (Sumithion 50EC) estimated from tomato.

Days after spraying	Sample weight (g)	Total volume prepared (ml)	Injected volume (µl)	Amount of Residue (mg/kg)	EU MRL (mg/kg)
0	10	10	1	8.983	0.01
1	10	10	1	6.846	
2	10	10	1	4.524	
3	10	10	1	2.464	
4	10	10	1	1.841	
5	10	10	1	0.765	
6	10	10	1	0.463	
7	10	10	1	0.205	
8	10	10	1	0.076	
9	10	10	1	ND	

Residue of fenitrothion in tomato was detected up to 8 DAS and these were 8.983 mg/kg, 6.846 mg/kg, 4.524 mg/kg, 2.464 mg/kg, 1.840 mg/kg, 0.765 mg/kg, 0.463 mg/kg and 0.205 mg/kg and 0.076 mg/kg at 0, 1, 2, 3, 4, 5, 6, 7 and 8 DAS, respectively. All of the samples had residues which were above MRL set by European Union. No residue was detected at 9 DAS. So, tomato can be harvested safely at 9 days after spray.

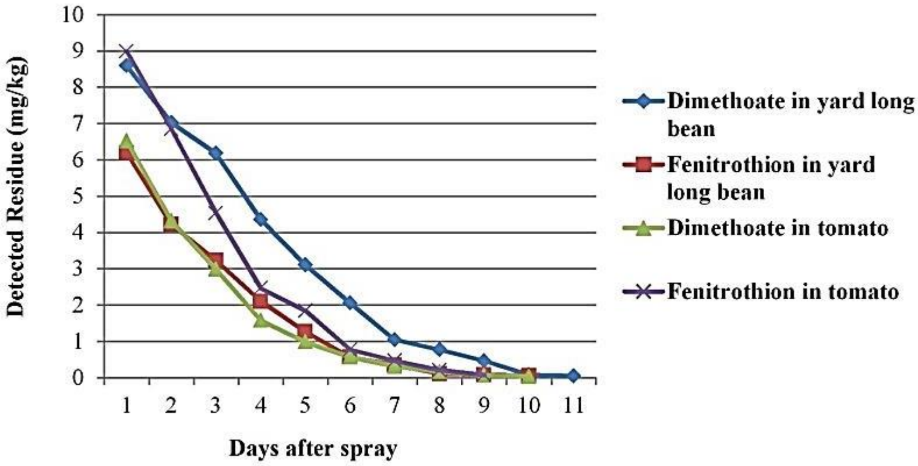

Figure 1. Dissipation of dimethoate and fenitrothion residues in yard long bean and tomato over time.

The trend of dissipation rate of dimethoate and fenitrothion residues in yard long bean and tomato over time are shown in Figure 1. It was observed from the figure 1, the dissipation rate of fenitrothion was little faster in tomato and slower in yard long bean and their residue levels were above MRL up to 8 DAS in tomato and 9 DAS in yard long bean. The rate of dissipation of dimethoate was one day letter in both the vegetables and the amount of their residues were above MRL up to 9 DAS in tomato and 10 DAS in yard long bean.

4. Discussion

The results of the present study showed that residue of dimethoate in yard long bean had been detected up to 10 DAS and in case of tomato it was 9 DAS. The quantities of residues were decrease down 7.021-0.046 mg/kg in yard long bean at 1-10 DAS and 4.319-0.034 mg/kg in tomato at 1-9 DAS which were above MRL set by European Union (2021). Residue of fenitrothion was detected up to 8 DAS in tomato and 9 DAS in yard long bean. The amount of residues were decrease down gradually 6.846-0.076 mg/kg at 1-8 DAS in tomato and 4.218-0.056 mg/kg in yard long bean at 1-9 DAS. All of the detected amounts were above MRL set by European Union. The rate of dissipation of the tested insecticides in both the vegetables was different. The plant behavior might be related to physic-chemical properties of pesticide, the dissipation of residue in and on plants is the effect of the interaction of environmental conditions such as wind, rain, sun, humidity, temperature and chemical and physical factor such as volatilization and growth of the plant (O' Brein, 1967; Virgina *et al.*, 1996; Jacobsen *et al.*, 2015).

Duara *et al.* (2003) found the level of residue of cypermethrin and fenvalerate below MRL at 7 DAS of recommended dose in brinjal. Sing and Kalra (1992) reported detectable level of residue of cypermethrin up to 7 DAS of prescribed dose in brinjal. Adnan *et al.* (2006) found diazinon residue above MRL up to 8 DAS in sweet pepper grown in green house. Ahmed *et al.* (2016) reported that the residue of fenvalerate was detected up to 14 DAS in hyacinth bean and tomato and the quantities were above MRL up to 3DAS. The residue of Acephate was detected up to 14 DAS in hyacinth bean and tomato but the quantities were above MRL up to 9 DAS because the dissipation rate of acephate was slow than fenvalerate in both the vegetables. Ahmed *et al.* (2020) found the dimethoate residue was detected up to 13 DAS in cauliflower, 12 DAS in hyacinth bean and 11 DAS in eggplant. Ahmed *et al.* (2021) also reported cypermethrin residue was detected up to 9 DAS in cauliflower, 7 DAS in brinjal and tomato and the residue was above MRL at 4 DAS in cauliflower and 3 DAS in brinjal and tomato. But in case of chlorpyrifosthe residue detected up to 7 DAS in brinjal and tomato and 9 DAS in cauliflower and all of the detected residues were above MRL. Prodhan *et al.* (2018) found diazinon, quinalphos and malathion residue in yard long bean was detected up to 10 DAS and the quantities of residue was above MRL at 5 DAS and the waiting period for tested pesticides in yard long bean was 7 DAS. The results of the present study accordant with the observation of the above authors.

Actually, in our country the farmers are not following the pre-harvest intervals (PHI), that is why, the residues of the applied pesticides are remaining in the harvested vegetables. There are several study (Habib *et al.* 2021; Hasan *et al.*, 2021; 2017; Islam *et al.*, 2021 ; Islam *et al.*, 2014; Nahar *et al.*, 2020; Parven *et al.*, 2021; Parvin *et al.*, 2021)) conducted with the most commonly used pesticides in the popular vegetables in Bangladesh prove that the farmers are not following the PHI. Therefore, the policy planners should take necessary steps to prevent this inappropriate practice to save the nation.

5. Conclusions

The dissipation rate of fenitrothion was little faster in tomato and slower in yard long bean. But in case of dimethoate it was one more day slower in both the vegetables. So, fenitrothion might be suitable insecticide for the vegetables which would be harvested at one day earlier than the vegetables sprayed with dimethoate. So, the safe period for fenitrothion in tomato and yard long bean was 9 and 10 DAS. In case of dimethoate it was 10 DAS in tomato and 11DAS in yard long bean. The findings of the study will help the crop growers, consumers and the other relevant stakeholders to conduct proper works for the production of safe food.

Acknowledgements

The authors are grateful to Md. Kamal Hossain in the Pesticide Analytical Laboratory, Pesticide Research and Environmental Toxicology Section, Division of Entomology, Bangladesh Agricultural Research Institute, Joydebpur, Gazipur-1701, Bangladesh for his kind cooperation during the study period.

Data availability

All the data presented in this study are contained in the manuscript.

Conflict of interest

None to declare.

Authors' contribution

Conceptualization and execution of study: Md. Sultan Ahmed; Methodology: Mohammad Dalower Hossain Prodhan, Md. Sultan Ahmed; Assisted in the laboratory works: Afroza begum and Marina Afroze; Writing-original draft preparation: Md. Sultan Ahmed; Writing-review and editing: Mohammad Dalower Hossain Prodhan, Nirmal Kumar Dutta and Debasish Sarker. All authors have read and approved the final manuscript.

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