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ESTIMATION OF RESIDUES OF FLUBENDIAMIDE AND DELTAMETHRIN IN CHICKPEA

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ABSTRACT

Persistence and residue study of flubendiamide and deltamethrin on chickpea pods and soil was carried out following foliar application of flubendiamide 90 + deltamethrin 60: 150 SC (W/V) @ 22.5 g a.i. ha^{-1} and @ 15.0 g a.i. ha^{-1}. In foliar application the initial residue of flubendiamide was found to be 0.61 and 1.35 mg kg^{-1} and deltamethrin to be 1.01 and 2.00 mg kg^{-1} at recommended and double the recommended dose, respectively. The residue dissipated below the limit of quantification at 7 and 10 for flubendiamide, 10 and 15 days for deltamethrin at recommended and double the recommended dose, respectively. The residue was below 0.05 mg kg^{-1} in chickpea pods and soil samples collected after 20 days of last application. Study of risk assessment revealed that the dose sprayed is completely safe, and waiting period of one day is to be observed.

Key words: Flubendiamide 90 + deltamethrin 60: 150 SC (W/V), dissipation, residue, pods, soil, GLC, half-life, HPLC, persistence, risk assessment, waiting period

Chickpea is a valued dietary crop (Wood and Grusak, 2007), and its nutritional value is well known as infant formula meeting the WHO/FAO requirements (Malunga et al., 2014). India is the single largest producer of chickpea with 65% share but it lacks behind in terms of productivity which is only 935.34 kg ha^{-1} (Merga and Haji, 2019). The productivity can be increased by diminishing the crop losses caused by insect pests. Major pests of the crop in India are the gram pod borer *Helicoverpa armigera* (Hubner), semilooper *Autographa nigrisigna* Walker, cut worm *Agrotis ipsilon* Hufnagel, termite *Odontotermes obesus* Rambor *Microtermes obesi* Holmgren, black bean aphid *Aphis fabae* Scopoli, white grub *Phyllopha gaimplicita* Horn, and tobacco caterpillar *Spodoptera litura* (F.) (Chandrashekhar et al., 2014). To prevent the damage, pesticide mixtures provide a promising option, and these broaden the activity spectrum overcoming pest resistance to single pesticide (Das, 2014). One such pesticide mixture is flubendiamide and deltamethrin.

The present study analyses the persistence, dissipation and risk assessment of flubendiamide and deltamethrin in chickpea. Flubendiamide is a novel systemic insecticide, highly effective for controlling lepidopteran pests (Nauen et al., 2007; Das, 2014; Ebbinghaus-Kintscher et al., 2007). A toxicologically important plant metabolite of flubendiamide is des-iodo flubendiamide which is formed as a result of loss of iodine present at 3-position of the phthalic acid moiety of the flubendiamide (EFSA 2013). Deltamethrin is type II pyrethroid with an α-cyano group, and it is a broad-spectrum insecticide (Tomlin 2006). Its mode of action is on the sodium ion channel (WHO Environmental Health Criteria, 1990), and it is registered for use on various crops including cereals, cotton, vegetables and field crops for pests such as aphids, mites, weevils, and beetles (Toxicological Profile for Pyrethrins and Pyrethroids, 2007). There are many reports regarding the residue studies of flubendiamide and deltamethrin present separately as single pesticide on many crops. Dissipation studies of flubendiamide were done by Mohapattra et al. (2010) in cabbage, Das et al. (2012) in okra and Takkar et al. (2013) in brinjal. Similarly for deltamethrin, dissipation studies were done by Kaur et al. (2011) in brinjal, Pandher et al. (2012) in chilli and Reddy and Reddy (2011) in cabbage. A few reports on combination formulations are also available. For eg., dissipation of flubendiamide and thiacloprid on tomato (Kooner et al., 2010), flubendiamide and thiacloprid residues in chilli (Parmar et al., 2012). But no literature is available on the combination formulation of flubendiamide and deltamethrin in chickpea. This study estimates the residues of flubendiamide and deltamethrin when applied as a mixed formulation of flubendiamide 90 + deltamethrin60 :150 SC (W/V) @ 22.5 g a.i. ha^{-1} and @15.0 g a.i. ha^{-1}.

MATERIALS AND METHODS

The certified reference standard of flubendiamide (purity 98.1 %), des-iodo flubendiamide (purity 99.8

%), and deltamethrin (purity 99.6 %) were procured from Bayer Mumbai. Solvents like acetone, chloroform, HPLC grade acetonitrile, sodium chloride were obtained from Merck. Anhydrous sodium sulfate and charcoal were taken from S D Fine Chemicals. Redistillation of various solvents was done using glass apparatus and reagent blanks were injected to check the purity of solvents and various other chemicals used during the processing. Flubendiamide and des-iodo flubendiamide standards of 1 mg ml^{-1} were prepared in acetonitrile and deltamethrin of 1 mg kg^{-1} was prepared in acetone: hexane mixture (1:1) and stored at 4°C. Intermediate working standard solution of 100 µg ml^{-1} was prepared and was used to prepare working standard solutions of various concentrations of flubendiamide, des-iodo flubendiamide and deltamethrin in respective solvents needed to construct a calibration curve (Fig. 1). The residues of flubendiamide and its metabolite des-iodo flubendiamide were quantified by HPLC (Shimadzu Company) having reversed-phase C_{18} column, photo diode array (PDA) detector and dual pump. HPLC operating conditions were as follows: Mobile phase: Acetonitrile: water: 70:30; flow rate of solvent: 0.3 ml min^{1} and wavelength of the PDA was 254 nm. By operating under these conditions, retention time of the flubendiamide and des-iodo flubendiamide were found to be 24.301 and 17.509 min. Analysis of the deltamethrin residues was done using gas liquid chromatograph (GLC- (Shimadzu Model GC-2010). A capillary column (30 mx 0.25 mm i.d) was used with the temperature programming of 280^{0}C for 5 min, followed by a rate of change of 5^{0}C min^{-1} to 230^{o}C for 20 min. The injector and detector were maintained at 280°C and 300°C, respectively. Carrier gas (N_2) flow was maintained @ 0.61 ml min^{-1}. The retention time of deltamethrin was 11.69 min.

Chickpea (variety PBG 5) was planted at Entomological Research Farm, PAU, Ludhiana, India. Field experiment was conducted using a randomized block design with three treatments pertaining to the residues of flubendiamide, des-iodo flubendiamide and deltamethrin in chickpea pods and soil. The treatment T_0 (control), T_1 (recommended dose (22.5 + 15 g a.i. ha^{-1}), and treatment T_2 (double the recommended dose (45 + 30g a.i. ha^{-1}) of the combination product (flubendiamide 90% + deltamethrin 60%), were made by applying 150 SC formulation @ 250 and 500 ml ha^{-1} in water (500 l ha^{-1}). The mixed formulation was sprayed thrice following good agricultural practices (GAP). The first spray was done at pod formation stage and subsequent ones at seven days interval as per the retreatment interval suggested for the field trials. Knapsack sprayer fitted with hollow cone nozzle was used for spraying. About half kg of chickpea pod samples were gathered

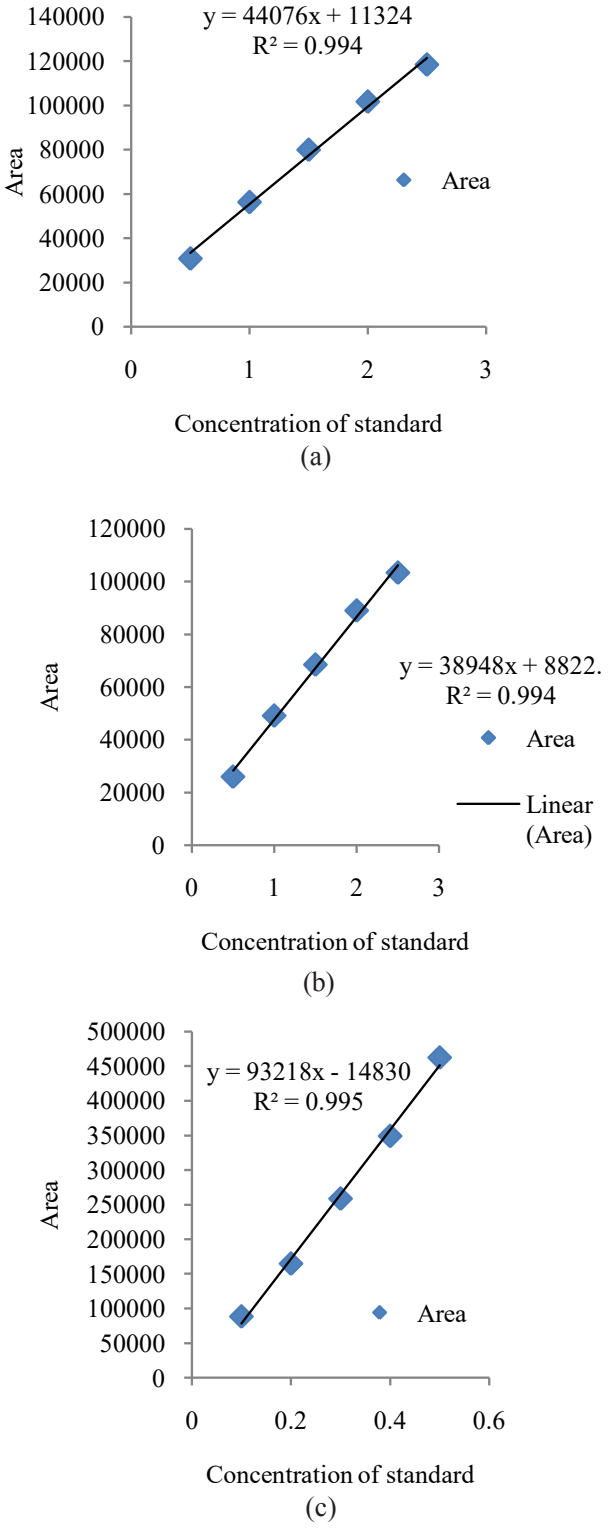


Fig. 1. Calibration curve of a) flubendiamide b) des-iodoflubendiamide c) deltamethrin

randomly from each treatment at 0 (2 hours), 1, 3, 5, 7, 10 and 15 days after third application. Mature seeds and soil samples were collected 20 days after third application. The chickpea pod samples from each treatment plot were mixed and sent to the laboratory where a representative sample of 50 g each was processed. Soil samples were taken from a depth of around 0-15 cm from 10 locales of each treated plot by using tube auger. After mixing and drying, the samples were sieved to remove any unwanted material. Moisture content of the soil samples was analyzed to get its dry weight for further calculations.

For extraction and clean-up of samples, methodology of Luke et al. (1975) was followed with few modifications. The methodology of partitioning was followed against QuEChERS methodology to achieve low limit of quantification value for the estimation of flubendiamide residues on HPLC. There were two setups for the extraction of residues. This is due to the different solubilities of flubendiamide and deltamethrin in different solvents. Flubendiamide and its metabolite dissolves in acetonitrile and deltamethrin in acetone. For flubendiamide- chickpea pods and soil (50 g each) were dipped separately into 100 ml acetonitrile and were kept overnight. The extract was filtered with the filter paper into a separatory funnel. It was further diluted with 600 mL brine solution, and then partitioned thrice with 100, 50 and 50 ml chloroform. The consolidated organic layers were passed through anhydrous sodium sulphate bolstered on glass wool in a filtering funnel. The extract obtained was concentrated to 25 ml in a rotary evaporator at 35°C. It was then treated with 500 mg activated charcoal powder and kept on a shaker for 2 hr. The clear extract was then filtered and concentrated and the last volume was made in acetonitrile. For deltamethrin, chickpea pods and soil (50g each) were dipped separately into 100 ml acetone and were kept overnight. The extract was filtered with the filter paper into a separatory funnel. It was further diluted with 600 ml brine solution, and then partitioned twice with 75 ml dichloromethane and twice with 75 ml hexane. Both the dichloromethane and hexane fractions were combined. The consolidated organic layers were passed through anhydrous sodium sulphate bolstered on glass wool in a filtering funnel. The extract obtained was concentrated to 3 ml in a rotary evaporator at 35°C. The extract was cleaned up using activated silica gel. A glass column was packed with 15 g of activated silica gel and mixed with 1.0 g of charcoal, in between two layers of anhydrous sodium sulphate supported on glass wool. The column was pre-washed with dichloromethane, and the concentrated extract was poured over it. The extract was eluted with a freshly prepared solvent mixture of dichloromethane and acetone (2:1, v/v). The clear extract was then filtered and concentrated to 2 ml in acetone.

RESULTS AND DISCUSSION

The instrumentation method used for the determination of residues of flubendiamide and its metabolite, des-iodo flubendiamide on HPLC and deltamethrin on GLC was validated in terms of its selectivity, linearity, precision in terms of repeatability and reproducibility and its limit of detection and quantification as per the SANTE guidelines (2015). Comparison of six control samples was made with that of six samples spiked at limit of quantification (0.05 µg ml^{-1}). No peak was found at the retention time of standard concerned in case of control samples. Hence, the selectivity of the method was checked. The matrix matched calibration curves of flubendiamide, des-iodo flubendiamide and deltamethrin were prepared to study the effect of matrix on the response of the analyte. Each produced a linear relationship with correlation coefficient (R^2) values above 0.990. The recovery studies were done at least three levels of fortification with LOQ as the lowest level of fortification. The acceptability criterion is the recovery range between 70-120%. Chickpea pods and soil samples were spiked with flubendiamide, de-iodo flubendiamide and deltamethrin at three levels of 0.05, 0.25 and 0.5 mg kg^{-1} and analysed as per the methodology described above. Recovery was >80 % in all the cases (Table 1). Therefore no correction factor was applied on the results obtained. Precision in terms of repeatability (RSD_r) of the method was determined by doing three replications of each fortification level. The acceptance criteria for RSD_r values is $\leq \pm$ 20 %. The RSD_r values are summarized in Table 1. Precision in terms of reproducibility (RSD_R) was checked by analyzing samples at LOQ level of 0.05 mg kg^{-1} for all the three pesticides under different set of conditions i.e. on different days or by different analysts. The acceptance criteria for RSD_R values is $\leq \pm$ 20 %. The RSD_R values are summarized in Table 1. The sensitivity of the detector for the analyte was calculated from limit of detection and limit of quantification values. For LOD calculations, the signal to noise (S/N) ratio was 3 and for LOQ the S/N value was 10.The limit of detection (LOD) and limit of quantification (LOQ) were worked out to be 0.016 and 0.05 mg kg^{-1} for all the three pesticides.

Dissipation trend of flubendiamide (mg kg^{-1}) on chickpea pods at various time intervals after the

Table 1. Recovery and RSD_r of flubendiamide, des-iodoflubendiamide and deltamethrin

Substrate	Level of fortification (mg kg^{-1})	Flubendiamide		Des-iodoflubendiamide		Deltamethrin	
		*Recovery (%)	RSD_r %	*Recovery (%)	RSD_r, %	*Recovery (%)	RSD_r, %
Chickpea pods	0.05	85.16± 2.48	2.91	86.37± 3.16	3.66	89.15± 2.18	2.45
	0.25	88.19± 2.38	2.70	88.49± 2.72	3.07	92.74± 3.65	3.94
	0.50	90.12± 3.15	3.50	89.61± 3.41	3.81	89.90± 3.18	3.54
Soil	0.05	92.17± 2.06	2.24	94.27± 2.15	2.28	92.32± 3.05	3.30
	0.25	94.36± 2.49	2.64	89.19± 2.65	2.97	95.08± 2.83	2.98
	0.50	90.75± 2.61	2.88	93.92± 2.39	2.54	89.71± 2.79	3.11

at 0.05 mg kg^{-1} level

Substrate	Day	Flubendiamide			Des-iodoflubendiamide			Deltamethrin		
		*Recovery (%)	RSD_r %	RSD_R %	*Recovery (%)	RSD_r, %	RSD_R %	*Recovery (%)	RSD_r %	RSD_R %
Chickpea pods	1	85.16± 2.48	2.91	3.04	86.37± 3.16	3.66	3.34	89.15± 2.18	2.45	2.93
	2	89.62± 2.79	3.11		92.08± 2.67	2.90		85.97± 1.94	2.26	
	3	90.27± 1.84	2.04		91.15± 2.23	2.45		85.06± 2.49	2.93	
Soil	1	92.17± 2.06	2.24	2.63	94.27± 2.15	2.28	3.06	92.32± 3.05	3.30	3.01
	2	90.16± 1.98	2.20		91.09± 3.76	4.13		89.43± 1.96	2.19	
	3	95.67± 2.76	2.88		91.46± 1.87	2.04		96.72± 3.38	3.49	

*Mean ± S.D. of three determinations

Residues of flubendiamide and deltamethrin (mg kg^{-1}) on chickpea pods and soil after foliar application of flubendiamide 90 + deltamethrin 60 :150 SC (W/V) @ 250 and 500 ml ha^{-1}.

Days after application	Flubendiamide				Deltamethrin			
	@ 22.5 g a.i. ha^{-1}		@ 45 g a.i. ha^{-1}		@ 15 g a.i. ha^{-1}		@ 30 g a.i. ha^{-1}	
	Mean± S.D.	% Dissipation	Mean± S.D.	% Dissipation	Mean± S.D.	% Dissipation	Mean± S.D.	% Dissipation
				Chickpea pods				
Before application	< LOQ		< LOQ		< LOQ		< LOQ	
0 (2 hrs after application)	0.61± 0.03	-	1.35 ± 0.03	-	1.01± 0.02	-	2.00 ± 0.03	-
1	0.52± 0.01	14.75	0.83 ± 0.04	38.52	0.85± 0.03	15.84	1.41 ± 0.17	29.50
3	0.29± 0.04	50.81	0.70 ± 0.02	48.15	0.65 ± 0.05	35.64	0.90 ± 0.02	55.00
5	0.23± 0.01	62.29	0.74 ± 0.04	74.07	0.44± 0.02	56.44	0.74± 0.04	63.00
7	< LOQ	-	0.43± 0.04	91.11	0.15± 0.04	85.15	0.43± 0.04	78.50
10	< LOQ	-	< LOQ	-	< LOQ	-	0.13± 0.03	93.50
15	< LOQ	-	< LOQ	-	< LOQ	-	< LOQ	-
				Mature pods				
20	< LOQ	-	< LOQ	-	< LOQ	-	< LOQ	-
				Soil				
20	< LOQ	-	< LOQ	-	< LOQ	-	< LOQ	-
$T_{1/2}$	2.87		3.14		3.38		2.61	

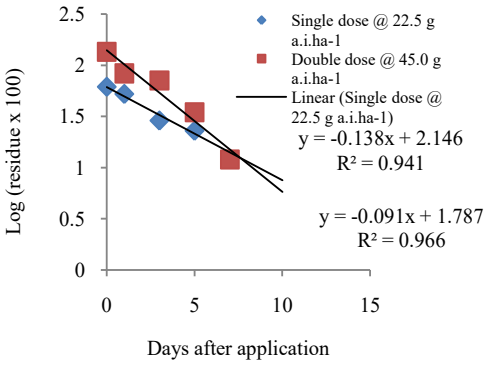
Maximum permissible intake (MPI) and theoretical maximum residue contribution (TMRC) of flubendiamide and deltamethrin in chickpea pods

Interval (days)	MPI (ug person^{-1} day^{-1})	Flubendiamide				MPI (ug person^{-1} day^{-1})	Deltamethrin			
		@22.5 g a.i. ha^{-1}		@45.0 g a.i. ha^{-1}			@15.0 g a.i. ha^{-1}		@30.0 g a.i. ha^{-1}	
		Residues (ug g^{-1})	TMRC (ug person^{-1} day^{-1})	Residues (ug g^{-1}	TMRC (ug person^{-1} day^{-1})		Residues (ug g^{-1})	TMRC (ug person^{-1} day^{-1})	Residues (ug g^{-1}	TMRC (ug person^{-1} day^{-1})
0	1200	0.61	0.73	1.35	1.62	1800	0.85	1.02	2.00	2.4
1	1200	0.52	0.62	0.83	1.00	1800	0.65	0.78	1.41	1.69
3	1200	0.29	0.35	0.70	0.84	1800	0.44	0.53	0.90	1.08
5	1200	0.23	0.28	0.35	0.42	1800	0.15	0.18	0.74	0.89
7	1200	<LOQ	-	0.12	0.14	1800	< LOQ	-	0.43	0.52
10	1200	<LOQ	-	<LOQ	-	1800	<LOQ	-	0.13	0.16
15	1200	<LOQ	-	<LOQ	-	1800	<LOQ	-	< LOQ	-

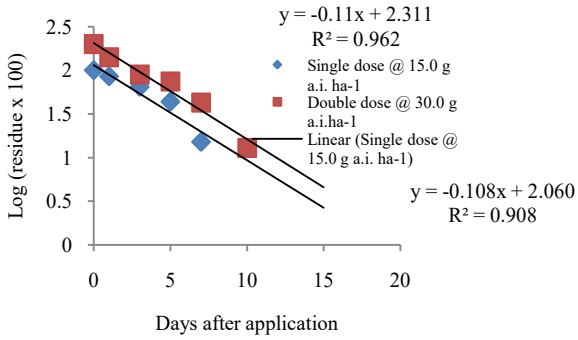
Limit of Quantification (LOQ) =0.05 mg kg^{-1}

application of the combined formulation @250 and 500 ml ha^{-1} representing recommended and double the recommended dose of 22.5 g a.i. ha^{-1}and 45 g a.i. ha^{-1} respectively, are presented in Table 1; initial deposits of flubendiamide on chickpea pods were calculated to be 0.61 mg kg^{-1}and 1.35 mg kg^{-1} at recommended and double the recommended dose, respectively. Residues dissipated to >50 % in both the dosages after 5 days. Residues of declined below the limit of quantification i.e. < 0.05 mg kg^{-1} at 7 and 10 days in two dosages. At harvest time of 20 days, none of the mature seeds and soil samples were detected for the presence of any residues. No residues of its metabolite des-iodo flubendiamide were found at LOQ level of 0.05 mg kg-1. The dissipation graph follows first-order kinetics (Fig. 2a). Half-life ($T_{1/2}$) were calculated as 2.87 and 3.14 at recommended and double the recommended dose, respectively. Dissipation trend of deltamethrin (mg kg^{-1}) on chickpea pods at various time intervals after the application of the combined formulation @250 and 500 ml ha^{-1} representing recommended dose of 15.0 g a.i. ha^{-1} and double the recommended dose of 30.0 g a.i. ha^{-1}, are presented in Table 1. Initial deposits were calculated to be 1.01 mg kg^{-1} at recommended dose and 2.00 mg kg^{-1} at double the recommended dose. Residues dissipated to more than 50 % after 5 days and declined below limit of quantification i.e. < 0.05 mg kg^{-1} at 10 and 15 days in both the dosages. At harvest time of 20 days, none of the mature seeds and soil samples were detected for residues. Half-life ($T_{1/2}$) of deltamethrin were calculated as 3.38 and 2.61, at recommended and double the recommended dose, respectively. Mukherjee et al (2015) calculated the dissipation trend of deltamethrin in a ready mix formulation of three pesticides on two crops i.e. tomato and egg plant when sprayed with 0.75% and1% deltamethrin at recommended and double the recommended dose of 1.0 and 2.0 l/ ha. Deltamethrin persisted till 5 days. Dissipation of deltamethrin followed first-order kinetics with half-life values ranged from 2.6 to 4.7 for tomato and egg plant, respectively (Fig. 2 b).

The consumption of food crops with pesticide residues may pose health hazards to the consumers if the residue levels in food commodity exceeds the maximum residue limit. MRL values are not available for the flubendiamide and deltamethrin in chickpea. Therefore theoretical maximum residues contribution (TMRC) were calculated and compared with maximum permissible intake (MPI) to evaluate the risk posed on consumer. Acceptable daily intake (ADI) is the amount



(a)



(b)

Fig. 2. Semi-logarithmic graph showing dissipation behaviour of a) Flubendiamide b) deltamethrin

of pesticide present in the daily diet of a person up to which it is safe and does not cause any health hazard upon consumption. ADI values for Flubendiamide and deltamethrin are 0.02 and 0.03 mg/ kg bw/ day, respectively. MPI was calculated as the product of average human weight (60 kg) and ADI. As per the National Sample Survey (Anonymous 2014), in rural areas, an average of 0.033 kg chickpea is consumed in 'other pulses' category in 30 days as compared to 0.036 kg/ 30 days in the urban area. Considering the bigger figure of 0.036 kg/ 30 days (1.2 g/ day) with the assumption that the entire commodity was contaminated with maximum amount of the pesticide residues. TMRC is obtained as the product of average daily consumption (g) and the residue levels of pesticide (ug g^{-1}) analysed a commodity. Table 1 depicts the comparison of the TMRC values with MPI in flubendiamide and deltamethrin, respectively. It was observed that TMRC values are far below the high values of MPI for both flubendiamide and deltamethrin. Study of risk assessment revealed that the dose sprayed is completely safe as even the highest residues detected for both the pesticides in 0 day are far below the maximum permissible intake (MPI). Therefore, following guidelines of good agricultural practices, waiting period of one day will be observed.

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