



DETERMINATION, DISSIPATION AND DECONTAMINATION OF THIAMETHOXAM USING LC-MS/ MS IN OKRA FRUITS

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ABSTRACT

An analytical method was developed and validated for the determination of thiamethoxam in okra using LC-MS/ MS. The LOQ was 0.002 $\mu\text{g g}^{-1}$. The recoveries at the fortification level of 0.002, 0.01, and 0.02 $\mu\text{g g}^{-1}$ were in the range of 75.45- 80.99% with an RSD of 3.54- 4.16%. Thiamethoxam applied at 25 (single dose) and 50 (double dose) g a.i. ha, recorded initial deposit of 1.541 and 3.117 $\mu\text{g g}^{-1}$ and half-life of 3.28 and 4.05 days were observed at single and double dose, respectively. The residue of thiamethoxam in fruits was reduced by simple decontamination methods to 20.34- 60.63%. Risk due to the dietary exposure was calculated for thiamethoxam and found safe on all the sampling days.

Key words: Thiamethoxam, okra, fruits, residue, decontamination, dissipation, LC-MS/ MS, recovery, single and double dose, half-life, dietary exposure

Okra *Abelmoschus esculentus* (L.) is an edible green nutritious seed pods vegetable, and about 72 insect pests are known to infest this. Among these, leafhopper (*Amrasca bigutulla bigutulla* Ishida) causes damage throughout the crop growth and causes yield loss; whitefly (*Bemisia tabaci* Gennadius) is another sucking pest and vector of vein clearing disease; and the shoot and fruit borer (*Earias vitella* F.) is a major fruit damaging pest, and collectively causing 36-90% yield loss (Gupta et al., 2009; Misra et al., 2002). For the control of these around 10 to 12 rounds of sprays are given leading to the problem of resistance, resurgence, residues, and decimation of useful fauna and flora (Dubey and Ganguly, 1998). Thiamethoxam is a neonicotinoid belong to the thianicotinyl subgroup with systemic and residual action and used against a wide range of insect pests (Maienfisch et al., 2001), and widely used as a foliar spray, soil drenching and seed treatment in vegetables against sucking pests (Ratnamma et al., 2020). Residue of this insecticides pose a potential risk to consumers because, it is not susceptible to ultraviolet light, degradation, ozonization (Ratnamma et al., 2021). Method validation for thiamethoxam in potato was studied using HPLC-DAD with the LOQ of 0.02 mg kg^{-1} (Abd-Alrahman, 2014). Method development and validation for the determination of neonicotinoids in leafy vegetables such as spinach are known. These provide recovery of 83% and LOQ of <1.4 $\mu\text{g/ kg}$ in LC-MS/MS (Pan et al., 2008) and 0.0015 to 0.008 and 0.005 to 0.025 $\mu\text{g/ g}$ LOQ and LOD in tomato,

respectively (Suganthi et al., 2018). Studies have reported the analysis of thiamethoxam and residues in okra, tomato, brinjal, mango (Singh and Kulshrestha, 2005; Hafez and Singh, 2016; Ramadan et al., 2016; Karmakar and Kulshrestha, 2009; Bhattacharjee and Dikshit, 2016). The removal of pesticide residue is essential and there is a need to decontaminate and made safe to the consumer with less labour (Subhash et al., 2014). Commonly vegetables are washed with water after they are brought from the market or field. Apart from this water washing, washing with several simple and cost-effective household methods, followed by boiling may reduce the residues. Taking these into account, the present study was undertaken to develop a method for the determination of residues, dissipation, and decontamination of thiamethoxam in okra.

MATERIALS AND METHODS

Reference standard of thiamethoxam (99.99%) was procured from Dr Ehrenstorfer, Augsburg, Germany. All the chemicals and reagents used were of analytical grade with > 99% purity. Stock solution (1000 $\mu\text{g/ ml}$) was prepared using pure certified reference material. The intermediate and working standard solutions were prepared through serial dilution techniques. The calibration standard ranging from 0.0005 to 0.100 $\mu\text{g/ ml}$ were prepared by dilution of stock solution with methanol. All the prepared standard solutions were stored at -20 °C and further used for method validation. LC-MS/MS model Shimadzu LC-MS 8040®

with column Shimpack XR, ODS III-C18, 2 mm id x 150 mm, ionization techniques- ESI, Column oven temperature- 40°C, polarity- +, injection volume 2 µl, injection rate 0.4 ml/ min, starting mobile phase B- 5.0%, nebulizing gas flow- 2.9 l/ min, drying gas- 15 l/ min, desolvation line temperature 250°C, ion source voltage 450°C, injection block temperature 250°C, heat block temperature 400°C, pressure limit (pump A, Pump B) Max 1300 Bar, Min 0 Bar, pump mode- binary gradient, and LC stop time of 3.5 min was used. QuEChERS method and its modification described by Anastassiades et al. (2003) and Lehotay et al. (2005) were adopted for extraction and cleanup of thiamethoxam in okra. Whole laboratory samples (500 g) were grounded thoroughly using a mixer grinder. About 10 g of grounded sample was weighed and transferred into a 50 ml centrifuge tube; 20 ml of acetonitrile was added and further allowed to stand for 30 min. The sample mixture was then homogenized at 10000-12000 rpm for 3 min. Then 3 g of NaCl was added and vortexed for 2 min. The homogenized sample mixture was centrifuged at 12000 rpm for 5 min. at 10 °C. After centrifugation, 15 ml of supernatant was collected in a test tube and added 9 g of Na₂SO₄. Further, 11 ml of extract was transferred from the test tube into a 15 ml centrifuge tube containing 0.4 g of primary secondary amine (PSA) and 1.15 g of MgSO₄ and then vortexed the mixture for 1 min. Centrifuge the supernatant with added reagent at 12000 rpm for 5 min. Then, 1 ml of supernatant was filtered using 0.22 µm PTFE nylon filter into LC vials. Identification and quantification of thiamethoxam in okra sample was optimized and validated according to the SANTE/11813/2017 (European Commission, 2017) by ascertaining the parameters such as; linearity, matrix effect, the limit of detection (LOD), the limit of quantification (LOQ), specificity, trueness, precision in terms of repeatability (RSD_r-intraday) and reproducibility (RSD_{wR}-interday).

Okra (var. Ankur-1) was grown at the Agricultural Entomology Block, UAS, Raichur in randomized block design. Two foliar sprays of thiamethoxam 25%WG at 25 and 50 g a.i./ ha as single and double dose, respectively at 15 days intervals during the fruit formation stage. Samples were drawn on 0 (2 hr after spray) 1, 3, 5, 7, 10, 15, 21, 25, 30, and 35 days after the second spray. The samples (500 g) were collected randomly from each treatment in a polythene bag and brought to the laboratory immediately for further processing and residue analysis (dissipation study). One day after spray samples were subjected to various decontamination methods given by the Rasolonjatovo

et al. (2017) and Aktara et al. (2010). The collected samples were extracted and cleaned up immediately after sampling on the same day; according to the modified QuEChERS method and injected into LC-MS/MS, calculated the residue of thiamethoxam. Waiting period and half-life was calculated following the procedures described earlier (Regupathy and Dhamu, 1990; Naik et al., 2020a, b). Hazard index (HI) was calculated by estimated average daily intake (EADI) from residues of thiamethoxam concentration (mg/ ks) obtained on different day samples and multiplied with per capita food consumption rate (kg/ day) for vegetables (357 g/ day) as per the procedure given by WHO (1997) and Gopalan et al. (1989).

RESULTS AND DISCUSSION

The acquisition parameters of the MS were optimized for the determination and quantification of thiamethoxam in okra. Initially, a full scan mass spectrum was recorded in array to choose the m/z value having the most abundance. For thiamethoxam, the parent ion (M+H)⁺ 292.00 was identified and selected as a precursor ion. Based on the known molecular ion, multiple reactions monitoring (MRM) transformation with different collision energies (CE) viz., -13, -24 and -21. The related acquisition condition with a binary gradient program of 3.50 min was standardized. The daughter ions of 211.10, 181.10, and 132.00 were selected for further quantification and confirmation, with ESI positive mode (Fig. 1). After determination of the MRM transitions, chromatographic conditions for better determination were found out; it was observed that the total ion chromatogram (TIC) had good separation resolution. The developed MRM positive mode gave more sensitiveness and accurate conditions for the detection at a low concentration of 0.0005 µg g⁻¹ in the matrix. Under the developed

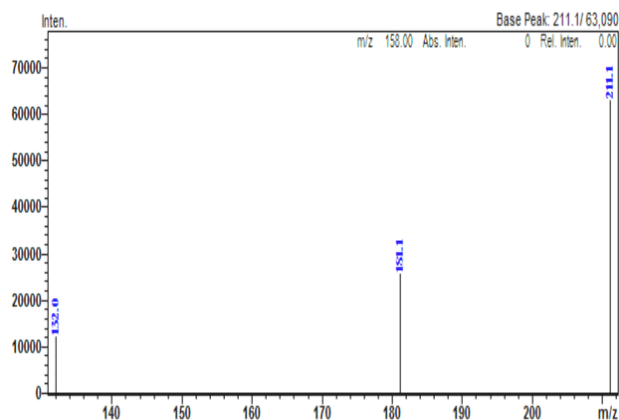


Fig 1. Product ion mass spectra of thiamethoxam

method thiamethoxam was found to elute at a retention time of 1.182 ± 0.1 min (Figs. 2, 3). The standardized method was validated according to SANTE/11813/2017 (European Commission, 2017) guidelines. Linearity for thiamethoxam was studied in okra matrix, as well as in methanol solvent in the calibration range of 0.001 to 0.1 $\mu\text{g/g}$. Good linearity and strong correlation between concentrations of peak area in terms of residuals were obtained at $\pm 20\%$ with the coefficient of determination (R_2) >0.998 . The LOD and LOQ in methanol solvent and okra matrix were selected (Table 1). The LOQ of the method compliance recovery of 70 to 120% was 0.002 $\mu\text{g/g}$, which is well below the MRL of 0.5 $\mu\text{g/kg}$ established by the European Commission.

The matrix effect calculated with the angular coefficient of the calibration curve was 18.26% which is in compliance with method validation criteria of $\pm 20\%$. The recovery was obtained at spiking levels of 0.002, 0.010, and 0.020 $\mu\text{g/g}$ and the mean recovery was found to be 80.99, 76.25, and 75.45%, respectively. Precision in terms of repeatability (RSD_r) and reproducibility (RSD_{wR}) was found between 70-120% spiked at the same level of recovery. RSD_r and % recoveries of thiamethoxam were 9.85, 4.55, and 4.16, and these were

found to be 86.97, 77.24, and 75.45% at a spiking level of 0.002, 0.01, and 0.02 $\mu\text{g/g}$, respectively. The inter day comparison of % RSD in terms of RSD_{wR} at the spiking level of 0.002, 0.010, and 0.020 $\mu\text{g/g}$ were 16.95, 7.90, and 7.89, respectively (Table 1). Earlier results reveal that recovery is in the range 71-119 and 70-120% in fruits and vegetables (Vidal et al., 2002; Carneiro et al., 2013) and pesticides were validated in fruits, vegetables, cereals, and pulses using LC-MS/ MS and GC-MS/ MS (Lehotay, 2005; Naik et al., 2021). The initial deposits of thiamethoxam were 1.541 and 3.117 $\mu\text{g/g}$ at single and double dose, respectively (Table 2). The residue of 1.93 and 3.71 $\mu\text{g/g}$ of thiamethoxam was observed for single and double dose, respectively on mango fruits (Bhattacharjee and Dikshit, 2016). Similarly, the initial residue of 1.62 mg/kg was observed on capsicum fruits applied at 48 g a.i./ ha (Pathipati, 2016). The residues of thiamethoxam dissipated from 70-77% after 3 days of the second spray and found accounting for the loss of 99-99.99% between 25-30 days. In other studies, it was observed that 100% loss of thiamethoxam takes place on 25 days after spray at 70 and 140 g a.i./ ha and the residue reached below the detectable limit at 15 days after spraying in tomato (Karmakar and Kulshrestha, 2009). The initial residue in okra dissipated to half of its concentration at 3.09 days at a single dose and 3.46 days at a double dose. In this study, prolonged dissipation behaviour of the thiamethoxam at both doses was observed.

All the decontaminating solutions significantly remove the residues of thiamethoxam in okra and the treatments differed in their efficiency ranging from 20 to 60%. Traditionally several simple methods were found effective for the removal of pesticide residues from the vegetables and safe to the consumers. The salt solution with boiling, tamarind solution, saltwater alone wash, washing with acetic acid were found to be effective. Dipping in 4% acetic acid was found to be significantly superior; dipping in 2% salt solution plus boiling for 15 min (57.11%), dipping in 0.1% sodium bicarbonate (54.63%), 2% tamarind (34.94%), 1% turmeric solution (32.73%), dipping in hot water (30.64%) and lemon solution (26.34%) were effective. The least reduction of thiamethoxam (20.34%) was observed by dipping in KMnO_4 solution (Table 2). Similar results from tap water as 36%, acetic acid, and potassium permanganate as 89%, and pressure cooking as 91 to 98% had been known (Tomer et al., 2014). Sodium bicarbonate (10%) and vinegar (10%) were found to be effective for dimethoate and acetamiprid residues, respectively, and thiamethoxam by both treatments

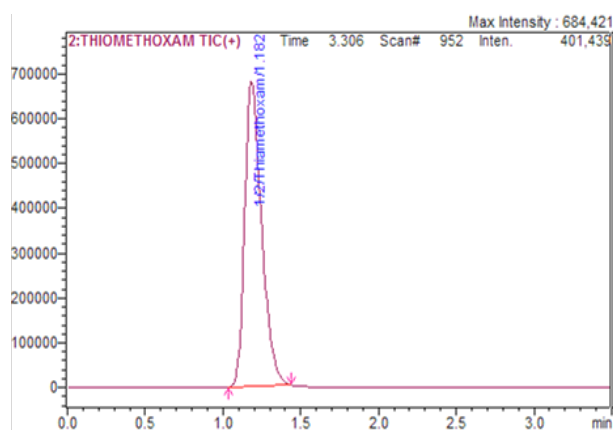


Fig. 2. Total ion chromatogram of thiamethoxam

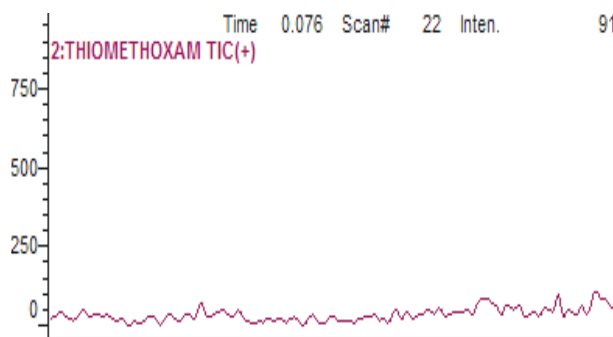


Fig. 3. The chromatograms represents the okra blank matrix free from thiamethoxam

Table 1. Linearity, detection, quantification and matrix effect of thiamethoxam in okra

	Regression equation	R ²	LOD	LOQ	Matrix effect (%)
Solvent	Y= 4500.8x-22339	0.999	0.0005 µg mL ⁻¹	0.0010 µg mL ⁻¹	-
Matrix	Y = 5322.8x-20619	0.999	0.0005 µg g ⁻¹	0.002 µg g ⁻¹	18.26

Accuracy			
Spiking level (µg g ⁻¹)	Mean recovery % (% RSD)	Precision in terms of repeatability % (% RSD)	Precision in terms of reproducibility % (% RSD)
0.002	80.99 (3.54)	86.97(9.85)	89.68(16.95)
0.01	76.25 (4.05)	86.97(4.55)	89.68(7.90)
0.02	75.45 (4.16)	86.97(4.16)	89.68(7.89)

R²- coefficient of determination; LOD- Limit of detection; LOQ- Limit of quantification; RSD- Relative standard deviation

Table 2. Residues of thiamethoxam and decontamination in okra (I day after application)

DAS	Residue at single dose		Residue at double dose	
	Residue (µg g ⁻¹) ± SD	% dissipation	Residue (µg g ⁻¹)± SD	% dissipation
0	1.541± 0.158	-	3.117± 0.134	-
1	0.767± 0.064	50.23	1.600± 0.091	48.67
3	0.387± 0.049	74.90	0.800± 0.049	74.33
5	0.199± 0.023	87.07	0.402± 0.140	87.11
7	0.096± 0.002	93.79	0.199± 0.025	93.63
10	0.048± 0.004	96.90	0.097± 0.010	96.89
15	0.024± 0.001	98.45	0.049± 0.004	98.43
21	0.010± 0.002	99.34	0.026± 0.004	99.17
25	0.003± 0.001	99.81	0.011± 0.003	99.65
30	BDL	-	0.007± 0.001	99.79
35	BDL	-	0.002± 0.001	99.89
Correlation coefficient	0.972		0.960	
Regression equation	y = 0.775 – 0.092x		y = 1.055 – 0.074x	
Degradation rate constant (day ⁻¹)	0.099		0.074	
Half life (days)	3.28		4.05	
Safe waiting period (days)	22.60		30.72	
Effect of decontamination				
Tr. No.	Treatment	Thiamethoxam		
		Residue (µg g ⁻¹)± SD	Reduction (%)	
T0	Control	0.767± 0.064	-	
T ₁	Dipping in 2 % tamarind solution for 15 min	0.499± 0.005	34.94 ^d	
T ₂	Dipping in 2 % salt solution for 15 min	0.321± 0.032	58.15 ^{a,b}	
T ₃	Dipping in 1 % turmeric solution for 15 min	0.516± 0.041	32.73 ^{d,e}	
T ₄	Dipping in 1 lemon in 1 L water for 15 min	0.565± 0.034	26.34 ^f	
T ₅	Dipping in 4 % of acetic acid solution for 15 min	0.302± 0.002	60.63 ^a	
T ₆	Dipping in 0.1 % KMnO ₄ solution for 15 min	0.611± 0.001	20.34 ^e	
T ₇	Dipping in 0.1 % of sodium bicarbonate solution for 15 min	0.348± 0.010	54.63 ^e	
T ₈	Dipping in hot water for 15 min	0.532± 0.009	30.64 ^e	
T ₉	Dipping in tap water for 15 min	0.521± 0.02	32.07 ^e	
T ₁₀	Dipping in 2 % salt solution for 15 min + Boiling for 15 min	0.329± 0.011	57.11 ^{b,e}	
S. Em±		0.55		
CD (p= 0.01)		1.64		
CV %		0.02		

DAS-Days after spraying, SD-Standard deviation

(Andrade et al., 2015). The maximum residue limit (MRL) of thiamethoxam on okra has been prescribed as 0.5 µg g⁻¹. The residues of thiamethoxam dissipated below the MRL on 10 and 21 days after application at single and double doses. The calculated hazard index based on the mean maximum concentration obtained in the respective treated dose was found to be <1 for all the days and samples in both the treated doses. These indicated that its safety for consumption and health of consumers (Table 3).

In this study, a very simple, robust QuEChERS method combined with LC-MS/MS was used for the determination of thiamethoxam. This method has considerable superiorities in respect of sample extraction and a short time program of analysis (3.5 min). The satisfactory LOQ (0.002 µg/ g) and accuracy (75.45-80.99%) demonstrate the suitability of the method. The residues of thiamethoxam were applied in an open field, recorded the initial deposit of 1.541 and 3.117 mg/ kg in single and double dose, respectively, and dissipated below its detectable in 25 (single dose) and 35 (double dose) days after the second spray. These insecticides need to be applied with caution with an adequate time gap before harvest to avoid detection of its residues at harvest. The safe waiting period will be useful to farmers to ensure safe consumption. Further, the decontamination study reveals that various household methods reduce the residue in the range of 20-60%.

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Table 3. Hazard index of thiamethoxam in okra

Days after treatment	Thiamethoxam							
	Mean residue (mg/ kg)	Recommended dose			Double the recommended dose			
		EDI	ADI ^b	HI ^c	Mean residue (mg/ kg)	EDI ^a	ADI ^b	HI ^c
0	1.541	0.009169	0.020000	0.458448	3.117	0.018546	0.020000	0.927308
1	0.767	0.004564	0.020000	0.228183	1.600	0.009520	0.020000	0.476000
3	0.387	0.002303	0.020000	0.115133	0.800	0.004760	0.020000	0.238000
5	0.199	0.001184	0.020000	0.059203	0.402	0.002392	0.020000	0.119595
7	0.096	0.000571	0.020000	0.028560	0.199	0.001184	0.020000	0.059203
10	0.048	0.000286	0.020000	0.014280	0.097	0.000577	0.020000	0.028858
15	0.024	0.000143	0.020000	0.007140	0.049	0.000292	0.020000	0.014578
21	0.01	0.000060	0.020000	0.002975	0.026	0.000155	0.020000	0.007735
25	0.003	0.000018	0.020000	0.000893	0.011	0.000065	0.020000	0.003273
30	-	-	-	-	0.007	0.000042	0.020000	0.002083
35	-	-	-	-	0.002	0.000012	0.020000	0.000595

EDI -estimated daily intake, ADI-acceptable daily intake, HI-hazard index

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