

Simultaneous Determination of Six Insecticides in Okra (*Abelmoschus esculentus* L. Moench) by UHPLC-MS/MS

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Abstract

The sensitive analytical method using quick, easy, cheap, effective, rugged and safe (QuEChERS) method for simultaneous determination of six insecticides in Okra was developed by UHPLC-MS-MS. The six insecticides were extracted from Okra matrices using acetonitrile and subsequently clean up using only octadecylsilane as sorbent prior to LC-MS-MS analysis. The determination of the target compounds were achieved in less than 2.0 min using an electrospray ionization source in positive mode (ESI+) for imidacloprid, acetamiprid, thiacloprid, thiamethoxam, clothianidin and negative mode (ESI-) for flonicamid. The method showed that the limits quantification (LOQ) ranged from 0.13 to 5.9 μgkg^{-1} in all matrices. The matrix-matched standard gave satisfactory recoveries (72.4%-105.1%) and relative standard deviation (2.2 - 20.0%) values in different matrices at three spiked levels (0.01, 0.1, and 1 mgkg^{-1}) for Okra.

Keywords: Residues analysis; Insecticides; Okra; Crop

Introduction

Pesticides, such as insecticides, herbicides, fungicides and acaricides, have been widely applied during the cultivation and the post-harvest storage of crops. These pesticides were used to prevent the destruction of edible crops by controlling agricultural pests or unwanted plants and thereby increases and improved food production [1].

Okra (*Abelmoschus esculentus* L. Moench) originated in Africa [2], is one of important vegetable crops and place in under Malvaceae family. Now it is growing in tropical and sub-tropical regions in the world. Okra is a nutritional vegetable, which it can promote gastrointestinal peristalsis and prevent constipation efficacy.

Acetamiprid a new generation from neonicotinoid insecticides and highly active to protect the various vegetable crops, by controlling mites and insect pests [3]. Imidacloprid is a systemic chloronicotinyl insecticide that enters the target pest via direct contact or ingestion. It is applied to seeds, soil, crops, and structures for controlling some insects. In addition, it can be used as a topical flea control treatment on domestic pets. Thiacloprid, a neurotoxic insecticide, belongs to family of the neonicotinoids [4]. Thiamethoxam is a second-generation neonicotinoid; it belongs to the thianicotinyl subclass that interferes with the nicotinic acetylcholine receptors in the insect's nervous system, it has systemic and residual activity in several crop plants against broad range pests [5]. Clothianidin, the newest member of the chloronicotinyl insecticide family, it has a high activity against a broad range of insects, including sucking insects, chewing insects, and some lepidopterans [6]. Fonicamid can be against to the aphids and other sucking insects and whiteflies, and other pests [7]. The Chemical structures of imidacloprid, acetamiprid, thiacloprid, thiamethoxam, clothianidin and flonicamid are represented in Figure 1.

The determination of these insecticides in the crops is very important to ensure food safety, and evaluate the risks posed by these insecticides to human, animals and environment. Some methods for determination of imidacloprid, individually in food and environmental matrices have been published using gas chromatography (GC and GC/MS) [8], Liquid chromatography-atmospheric pressure chemical ionization-mass spectrometry (LC-APCI-MS) [9], liquid chromatography-mass

spectrometry (LC-MS) [10-13]. Imidacloprid and acetamiprid were analyzed by high performance liquid chromatography (HPLC) [14-16]. Others some reports for determination analysis of acetamiprid, Thiacloprid, Thiamethoxam, flonicamid and clothianidin in different matrices can be found by the HPLC [17] to analysis acetamiprid by liquid and gas chromatography coupled to mass spectrometry for determination acetamiprid, clothianidin, thiacloprid [18]. Some also reported for analysis of thiacloprid, the ion chromatography (IC) [19]. There are some reports have been made for the analysis of thiamethoxam by HPLC with an electrochemical detector and post-column photochemical reactor [20]. Thiamethoxam and Clothianidin by Using GC with- μECD or HPLC-UVD [21,22]. A few reports have made for analysis in food, such as analysis flonicamid in dried hops by liquid chromatography (LC) tandem mass spectrometry (MS/MS) [23], simultaneous determination of flonicamid and its metabolites in vegetables using QuEChERS and reverse-phase liquid chromatography-tandem mass spectrometry [24]. There are simultaneous determinations of five insecticides in bees (acetamiprid, thiacloprid, thiamethoxam and clothianidin) by LC-MS/MS QuEChERS [16-25].

The LC-MS/MS has already been proved powerful and widely used technique. Thanks to progress of the chromatography technique, ultra-high-performance chromatography (UHPLC) was developed by using columns containing particles with a diameter of $<2 \mu\text{m}$ than conventional LC and fluidic systems that operate at higher pressures, resulting in shorter analysis time and increase the peak resolution, capacity, and sensitivity [26].

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This paper described a simultaneous determinations analysis of six insecticides (acetamiprid, imidacloprid, thiacloprid, thiamethoxam, flonicamid and clothianidin) were extracted from Okra matrices using acetonitrile and subsequently clean up using only octadecylsilane (C_{18}) as sorbent prior to LC-MS-MS analysis. To our knowledge, this is the first report to establish an analytical method for determination of six insecticides (acetamiprid, imidacloprid, thiacloprid, thiamethoxam, flonicamid and clothianidin) in Okra using UHPLC-MS/MS. The developed method was also validated by application to the analysis sample.

Materials and Methods

Reagents and materials

Analytical standards of imidacloprid (99.9% purity), clothianidin (99.8%) and thiamethoxam (99% purity) were bought from Agro-Environmental Protection Institute, Ministry of Agriculture (Tianjin, China). Acetamiprid (97.2% purity) and thiacloprid (99% purity) were supplied from Bayer Crop Science, (Frankfurt, Germany), Flonicamid

(100% purity) was obtained from Ishihara sangyoKaisha. ISK (Beijing, China). Analytical grade methanol, acetonitrile for pesticides residue analysis were purchased from Beihua fine-Chemical Co. (Beijing-China). Acetonitrile and formic acid (chromatography grade) were obtained from Fisher Scientific (New Jersey, USA). Anhydrous Magnesium Sulfate ($MgSO_4$) and (NaCl) was purchased from Sinopharm Chemical Reagent Co. Ltd (Beijing-China). The octadecylsilane (C_{18} , 40 μm) sorbets purchased from Angela Technologies Int. (Tianjin, China). Purified water was prepared by using Milli-Q water purification system (Millipore Purification Systems).

Preparation of standard solution

The stock standard solution of imidacloprid (100 mg L^{-1}), acetamiprid (100 mg L^{-1}), thiacloprid (100 mg L^{-1}), clothianidin (100 mg L^{-1}), thiamethoxam (100 mg L^{-1}) and flonicamid (100 mg L^{-1}) were prepared in acetonitrile. This solution was diluted to obtain 10.0, 1.0, 0.5, 0.1, 0.05, 0.01 mg L^{-1} in acetonitrile. All solution was stored in a refrigerator in the dark at 4°C and the working standard solutions underwent

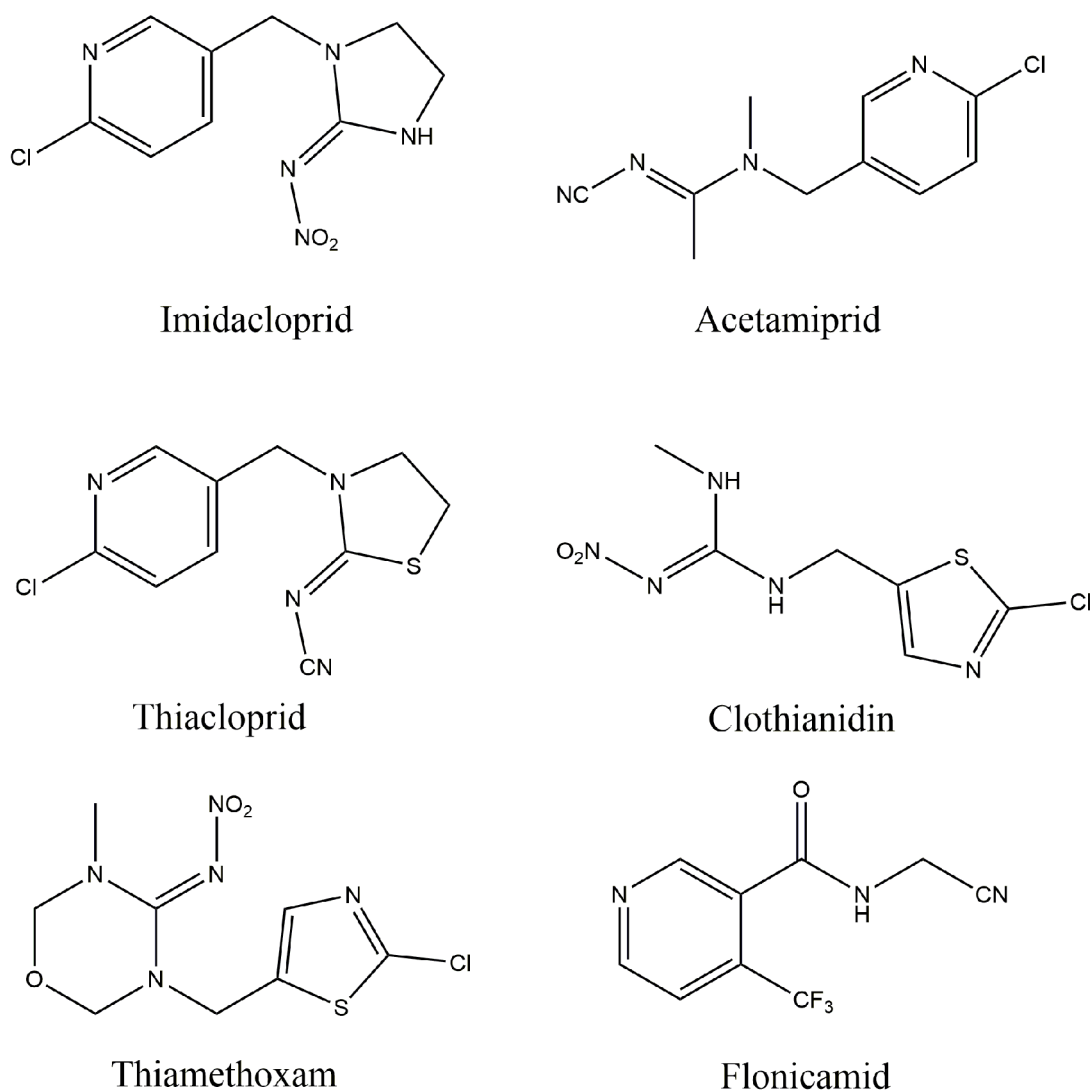


Figure 1: The Chemical structures of imidacloprid, acetamiprid, thiacloprid, thiamethoxam, clothianidin and flonicamid.

no degradation for 6 months. The Okra (*Abelmoschus esculentus L. Moench*) was obtained from market in Beijing. The matrices were not applied or contaminated by six insecticides, and they were put into polyethylene bags. They were transported to the laboratory and stored in the dark at less than -18°C until analysis.

Instrumentation and chromatography conditions

Extraction equipment: The mobile phase solvent were distilled and passed through a $0.22\ \mu\text{m}$ pore size filter before use. Purified water obtained by purifying demineralized water in Milli-Q Integral 3 water (Millipore, Bedford, MA, USA), a shaker (model HZO-CA, Jintan, China) and centrifuge (model Excelsius II, Fanen, Brazil) were used for sample preparation. An octadecylsilane (C_{18}) column and a $0.22\ \mu\text{m}$ obtained from Agela Technologies (Tianjin, China).

Apparatus and chromatography: Chromatographic separation for six insecticides was performed on a Waters (Milford, MA, USA). ACQUITY UPLC system equipped with a Waters ACQUITY UPLC BEH bridged ethylene hybrid C_{18} column ($50\ \text{mm} \times 2.1\ \text{mm}$, $1.7\ \mu\text{m}$ particle size) (Milford, MA, USA). The mobile phase of acetonitrile (solvent A) and 0.1% (v/v) formic acid in water (solvent B), were pumped at a flow rate of $0.3\ \text{mL min}^{-1}$. Simultaneous separations were completed using a gradient profile of $0.0\ \text{min}/10\% \text{ A}$, $1.5\ \text{min}/70\% \text{ A}$, $2.5\ \text{min}/90\% \text{ A}$, $2.6\ \text{min}/10\% \text{ A}$, and $4.5\ \text{min}/10\% \text{ A}$, respectively. The injection volume was $3\ \mu\text{L}$. The column was kept at 40°C to decrease the viscosity, and the temperature in the sample manager was set at 5°C . All six Insecticides were eluted within 2.0 min.

Analysis of insecticides (Imidacloprid, acetamiprid, thiacloprid, thiamethoxam, clothianidin and flonicamid) compounds was conducted on a triple-quadrupole mass spectrometer (TQD, Waters Crop.) equipped with electrospray ionization source (ESI). The nebulizer gas was 99.95% nitrogen, and the collision was 99.999% argon with a pressure of 2×10^{-3} mbar in the T-wave cell. MS/MS detection was performed in positive and negative ion mode and the monitoring conditions were optimized for target compounds. The conditions were typically as follows: The capillary voltage was set at $3.0\ \text{kV}$, while the source temperature and desolvation temperature were held at 120°C and 350°C , respectively. A $50\ \text{Lh}^{-1}$ cone flow and $500\ \text{Lh}^{-1}$ desolvation gas flow were used respectively. Multi-reaction monitoring (MRM) was

used for the detection of all compounds with a dwell time of $0.05\ \text{ms}$. Infusion experiments of each compound were conducted to optimize the intensity in both positive and negative ionization modes. All other ESI and MS parameters were optimized individually for each target insecticide and were listed in Table 1. The Masslynx NTV.4.1 (Waters, USA) software was used to collect and analyze the data obtained.

Sample preparation: Approximately $500\ \text{g}$ of Okra samples were chopped and homogenized by Ultra-Turrax T25 Mixer at $9500\ \text{rpm}$. In total $5\ \text{g}$ aliquots homogenized samples were weighed into $50\ \text{mL}$ centrifuge tube, $5\ \text{mL}$ acetonitrile and $3\ \text{mL}$ of water were added on okra. The samples in tubes were shake vigorously for $3\ \text{min}$ by using vortex mixer to ensure that the solvent interacted well with the entire samples. The samples tubes were then stored in a refrigerator at -20°C for $30\ \text{min}$, after that $3\ \text{g NaCl}$ and $2\ \text{g MgSO}_4$ were added to the sample in the tube and vortexed immediately for $1\ \text{min}$ and then the extracts were centrifuged for $5\ \text{min}$ at $5000\ \text{rpm}$. A volume of $2\ \text{mL}$ prepared aliquot sampled from upper layer into another $5\ \text{mL}$ centrifuge tube containing $50\ \text{mg}$ only C_{18} use it as sorbent. All the samples vortexed again for $1\ \text{min}$ and then centrifuged for $5\ \text{min}$ at $5000\ \text{rpm}$. After that $1.5\ \text{mL}$ of upper layer was filtered using $0.22\ \mu\text{m}$ nylon syringe filter and transferred into an auto-sampler vial for chromatography injection at LC-MS/MS.

Method validation: Blank samples of Okra were analyzed to verify the absence of interfering species at about the retention time of the analytes. The linearity of the method was studied by analyzing different matrix – matched standard solutions in triplicate at five concentrations ranging from 0.01 to $1\ \text{mg L}^{-1}$. The parameter of linear regression equations, the standard deviation and the correlation coefficient (R^2) were calculated in Table 2. Precision (when repeated independent analysis was performed), accuracy (when recovery assays were performed), sensitivity.

The matrix-dependent limits of quantitation (LOQ) and limit of detection (LOD) were calculated for the analytical methodology using the blank and calibration standards of Okra. The LODs of six insecticides are the concentrations that produce a signal to noise (peak to peak) ratio of 3. The LOQs are defined based on signal-to-noise ratio of 10. The LOQs are estimated from the chromatogram corresponding

!Compound	Molecular formula	MW	t_r (min)	Ion Source	CV (V)	Quantification ion transition	CE 1 (eV)	Diagnostic ion transition	CE 2 (eV)
Thiamthoxam	$\text{C}_8\text{H}_{10}\text{ClN}_5\text{O}_3\text{S}$	291.7	1.11	ESI+	35	2 92→211	18	292→133	20
Imidacloprid	$\text{C}_9\text{H}_{10}\text{ClN}_5\text{O}_2$	255.6	1.21	ESI+	30	2 56→209	18	256→175	20
Clothianidin	$\text{C}_6\text{H}_8\text{ClN}_5\text{O}_2\text{S}$	249.6	1.19	ESI+	24	2 52→126	18	250→90	30
Thiacloprid	$\text{C}_{10}\text{H}_9\text{ClN}_4\text{S}$	252.7	1.36	ESI+	18	2 50→169	18	252→90	30
Acethamprid	$\text{C}_{10}\text{H}_{11}\text{ClN}_4$	222.6	1.27	ESI+	20	223.2→207	20	223.3→126	20
Flonicamid	$\text{C}_9\text{H}_6\text{F}_3\text{N}_3\text{O}$	229.2	1.14	ESI-	10	228→188	10	228→81	10

MW: Molecular Weight; CV: Cone Voltage; CE: Collision Energy; Ion ratio: Area of qualitative ion/area of quantification ion

Table 1: Experimental parameters and UHPLC-MS/MS conditions of the six compounds in ESI+ and ESI- mode.

Compound	Matrix	Regression equation	R^2	LOQ ($\mu\text{g}/\text{kg}^{-1}$)	LOD ($\mu\text{g}/\text{kg}^{-1}$)
Thiamethoxam	Okra	$y=8.8804x+180.91$	0.9882	1.9	0.12
Imidacloprid	Okra	$y=25855x+5974.7$	0.9994	0.2	0.1
Clothianidin	Okra	$y=18499x-3.6535$	1	1.7	0.5
Acetamiprid	Okra	$y=8.6611x-45.193$	0.9996	2.2	0.5
Flonicamid	Okra	$y=38033x-825.45$	0.9984	0.13	0.09
Thiacloprid	Okra	$y=4018.3x-5.9251$	0.9961	5.9	2.5

Table 2: Comparison of matrix-matched calibration and solvent calibration at $10\text{-}1000\ \mu\text{g}/\text{L}$.

to the lowest point used in matrix matched calibration. Recoveries were determined for five replicates of the spikes samples at different three levels of each insecticide for Okra with standard working solutions. The spiked samples were allowed to equilibrate for 1 h prior to extraction.

Results and Discussion

Optimization of chromatography

The optional separation conditions including different mobile phase compositions (ACN/water, Acetonitrile, Water, ACN/1.0% formic acid aqueous solution) were established by injecting 3 μ L of six insecticides mixture standard solution. The six insecticides were separated using Acetonitrile-water as shown in Figure 2. There were no interference peaks in region of chromatography and analysis time of the six insecticides was less than 2 min. UHPLC was performed using BEH C_{18} column (50 mm \times 2.1 mm, 1.7 μ m particle size) in this study allowed a considerable reduction of LC analysis (6.39 to 10.57 min) (Kamel, 2010), and (10 to 15 min) for acetamiprid, imidacloprid [15].

Optimization of MS/MS

Full-scan and MS/MS mass spectra were obtained from the infusion of 5 mg L^{-1} standard solution of these compounds in acetonitrile-water at a flow rate of 10 $mL\ min^{-1}$. The analysis of six insecticides was performed in MRM mode, to optimize MS/MS conditions of mass spectrometry and the target compounds presented and achieving a compromise between both positive and negative modes, for acetamiprid, Imidacloprid, Thiacloprid, Thiamethoxam, and clothianidin, in this study, ESI in positive mode was selected for subsequent experiments for the results demonstrated higher responses in positive mode than in negative mode. The chemical formulas, molecular weights, cone voltages, precursor ions, and collision voltages were also listed in Table 1.

As shown in Figure 2, six insecticides could be detected at the spiked level. When a non-spiked sample was also subjected to the entire procedure, no interfering peaks were observed in any of the samples. As shown in Table 2, the LODs for the six pesticides were estimated to be 0.09-2.5 μ g kg^{-1} , and the LOQs for six insecticides were 0.13-5.9 μ g kg^{-1} .

Precision and accuracy

Recovery studies were performed to validate the UPLC-MS/MS method by spiking the blank samples at three different concentration levels (0.01, 0.1, and 1 $mg\ kg^{-1}$) in Okra, and then analyzed in quintuplicate. The recoveries were calculated using the three-point matrix matched calibration curves presented in Table 3. Only one type of sorbent C_{18} is used in this work to investigate the influences on recovery rate in two matrices. The C_{18} is suitable to extract non-polar and medium-polar compounds from the polar samples, which are mainly used for antiphase extraction. The precision of the method was determined by the repeatability and reproducibility and expressed by the relative standard deviations (RSDs). The intra-day precision was measured by comparing the standard deviation of the recovery percentages of spiked samples run during the same day. The inter-day precision was determined by analyzing spiked samples for three distinct days.

In general, the mean recoveries ranged between 72.4% and 105.1% for the spiked levels (Table 3). And the RSD^b (inter-day precision) for the proposed method were ranged from 9.1%-19.6%. These results showed that the developed residue method was satisfied for all six target compounds in Okra matrices.

Method application

In order to demonstrate the applicability of this method for

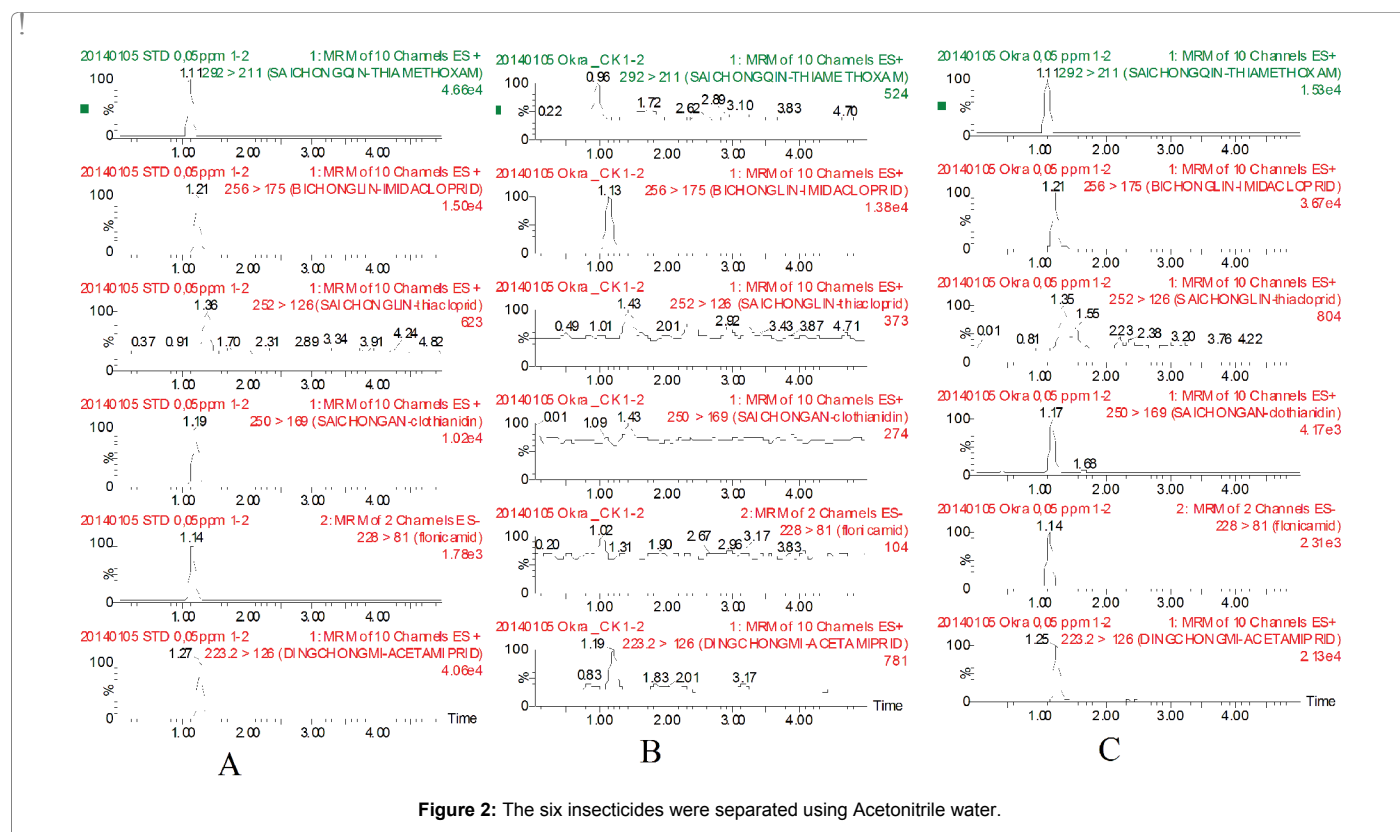


Figure 2: The six insecticides were separated using Acetonitrile water.

Matrix	Spiked level (mkg ⁻¹)	Thiamethoxam			Imidacloprid			Thiacloprid		
		Recovery	RSD ^a	RSD ^b	Recovery	RSD ^a	RSD ^b	Recovery	RSD ^a	RSD ^b
Okra	0.01	79.6	4.7	10.0	75.7	12.8	13.0	79.5	11.5	10.8
	0.1	79.8	9.2	11.2	105.1	8.1	14.1	100.2	3.8	18.0
	1	76.1	2.4	10.7	81.0	3.2	9.1	78.3	2.2	9.4

Matrix	Spiked level (mkg ⁻¹)	Clothianidin			Acetamiprid			Fonicamid		
		Recovery	RSD ^a	RSD ^b	Recovery	RSD ^a	RSD ^b	Recovery	RSD ^a	RSD ^b
Okra	0.01	78.1	20.0	19.6	104	14.8	18.4	88.8	11.5	10.2
	0.1	91.8	8.7	12.8	95.5	15.8	10.8	81.4	9.4	13.1
	1	73.7	4.8	12.2	91.7	7.2	17.1	72.4	2.4	15.5

^aIntra-day (n=5); ^bInter-day (n=15)

Table 3: Recoveries (n=15, %) and RSD (%) for target compounds from different matrices in three spiked levels.

monitoring of this pesticides residue in Okra. Five okra samples were obtained from market in Beijing and randomly was analyzed. The samples were treated with the sample preparation method described in section 4.3.3. All samples and did not represent a threat for the consumer since they were not detected any residue and below the MRLs established by America (0.4-1 mg kg⁻¹) and Japan (0.7-5 mg kg⁻¹).

Conclusions

This work described the development and validation a simple LC-MS/MS method for the simultaneous determination of residues of six insecticides (Imidacloprid, acetamiprid, thiacloprid, thiamethoxam, clothianidin and fonicamid) in Okra. Extracts containing the target compounds were analyzed and validated by UHPLC-MS/MS. This method allowed separation of the six target pesticides in less than 2 min with good specificity. The recovery percentages were ranged from 72.4% to 105.1% with RSD in the range of 2.2% to 20.0% for all analytes in okra matrices. In addition, the specificity, calibration curves, precision, and reproducibility were tested successfully. As a conclusion, the proposed method is easy and useful in analysis these six insecticides. This method also can be recommended for monitoring studies in the okra to ensure food safety.

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