Agricultural Sciences, 2014, 5, 850-857
Published Online August 2014 in SciRes. http://www.scirp.org/journal/as
http://dx.doi.org/10.4236/as.2014.510091

Degradation Dynamics and Residue Analysis of Flubendiamide in Cabbage and Soil by Liquid Chromatography-Tandem Mass Spectrometry with Dispersive Solid Phase Extraction

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Received 22 June 2014; revised 24 July 2014; accepted 6 August 2014

Abstract

To formulate a scientific basis for a reasonable spray dose and safe interval period of 20% flubendiamide water dispersible granule (WDG) on controlling vegetable pests, degradation dynamics of flubendiamide in cabbage and soil was analyzed in this study. Dissipation and residue of flubendiamide in 20% flubendiamide WDG in cabbage and soil under field conditions were investigated by liquid chromatography-tandem mass spectrometry with dispersive solid phase extraction. Results showed that the degradation dynamic equations of flubendiamide in cabbage and soil were based on the first-order reaction dynamic equations. The half-lives of the degradation of flubendiamide were 3.51 d to 3.96 d and 3.43 d to 3.87 d in the cabbage of Yangzhou and Jingzhou, respectively, and 4.42 d to 5.13 d and 4.37 d to 4.99 d in the soil of Yangzhou and Jingzhou, respectively. The terminal residues of flubendiamide in the cabbage of Yangzhou and Jingzhou were 0.0247 mg·kg^{-1} to 0.0393 mg·kg^{-1} and 0.0225 mg·kg^{-1} to 0.0273 mg·kg^{-1}, respectively, when 20% flubendiamide WDG was applied at a dose of 0.050 g·m^{-2}. Flubendiamide is safe to be applied in cabbage fields at the recommended dose.

Keywords

Flubendiamide, Dispersive Solid Phase Extraction, LC-MS/MS, Degradation, Residue

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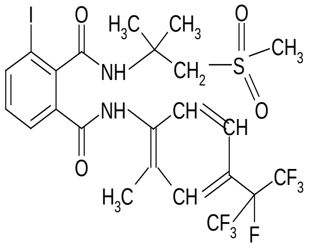
How to cite this paper: Chen, X.J., Wang, P., Meng, Z.Y., Chen, S., Gu, H.T. and Sha, X.L. (2014) Degradation Dynamics and Residue Analysis of Flubendiamide in Cabbage and Soil by Liquid Chromatography–Tandem Mass Spectrometry with Dispersive Solid Phase Extraction. *Agricultural Sciences*, **5**, 850-857. http://dx.doi.org/10.4236/as.2014.510091

1. Introduction

Flubendiamide is an inhibitor of ryanodine receptor that was developed by the Japanese Pesticide Corporation, Ltd. and first registered in the Philippines in 2007 [1] [2]. Flubendiamide is mainly effective for controlling lepidopteron pests, including resistant strains in vegetables, fruits, rice, and cotton. This insecticide has good, rapid, and long-term activity against both adults and larvae. Flubendiamide is a new phthalic amide insecticide that has a function in the activation mechanism of intracellular calcium release channels (ryanodine receptors), a leading cause of uncontrolled storage of calcium release. Given the uniqueness of this mechanism, no cross-resistance to conventional insecticides has been found, and it is safe for arthropods and mammals [1] [3]-[6]. The 20% flubendiamide water dispersible granule (WDG) is registered for controlling vegetable insects, such as *Spodoptera exigua* or *Plutella xylostella*, in China. In some reports, flubendiamide WDG is used to control rice pests. Flubendiamide is retained in cabbage after application in the field, and enters the soil and water system through a variety of ways. To date, reports on the detection and analysis methods of flubendiamide focus on the analysis of flubendiamide using HPLC-UV (UV-visible detector) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) ion switching in the ESI position mode [7]-[14].

Field dissipation studies on pesticide persistence in foodstuff and behavior of pesticide residues in agricultural fields are necessary to ensure food safety and protection of the environment [15]. This study used the method based on LC-MS/MS in the ESI negative mode coupled with dispersive solid phase extraction method for identification and quantization of flubendiamide residues in 20% flubendiamide WDG in cabbage and soil under field conditions. Experimental trials were conducted to determine and evaluate flubendiamide residue levels and dissipation rates in cabbage and soil under field conditions. The results can provide basic information to develop regulations on the safe use of flubendiamide in cabbage fields and to prevent any consumer health problems.



Chemical structure of flubendiamide.

2. Experimental Section

2.1. Equipments and Chemicals

The instruments were BS210S electronic balance, SB-1000 rotary vacuum evaporator and THZ-82A vibrating machine made by Sartorius company of Germany, Eyela company of Japan and FuHua Company of Jiangsu province, China, respectively. Agilent 1200 Series HPLC system equipped with an Agilent 6460 Triple Quadrupole LC/MS system was made by Agilent Co. (USA).

Flubendiamide (purify 98.52%) and flubendiamide WDG (20%) were purchased from Fluka Company (USA) and Nihon Nohyaku Co. Ltd. (Japan), respectively. Dehydrated magnesium sulfate ($MgSO_4$), Primary secondary amine (PSA), and C_{18} were obtained from DIMA Technology, Inc., USA, and all of those were analytical grade. Methanol and acetonitrile were obtained from DIMA Technology, Inc., USA, and both were HPLC grade.

2.2. Experiment Methods

2.2.1. Field Trials

Field trials were conducted in Yangzhou City and Jingzhou City, China in 2011 and 2012. The tested plants were cabbage and its variety was Jiangfeng NO. 1. These trials were in accordance with the Guidelines on Pesticide Residue Trials [16] issued by the Ministry of Agriculture, People's Republic of China.

During the experimental period in 2011and 2012, rainfall events did not occur in Yangzhou and Jingzhou. In 2011, the average temperatures in Yangzhou and Jingzhou were 27.3˚C and 28.1˚C, and the corresponding values for 2012 were 27.9˚C and 28.3˚C, respectively. The experimental sites exhibit distinguishable soil properties.

For example, the soil of Yangzhou is sandy clay loam with 18.5 g·kg^{-1} of organic matter and pH of 6.7, and the soil of Jingzhou is clay loam with 20.1 g·kg^{-1} of organic matter and pH of 6.5.

Each experimental treatment consisted of three replicates and a control plot. A buffer area of 15 m^2 separated each plot. To study flubendiamide dissipation in cabbage and soil, 20% flubendiamide WDG was applied at 0.05 g·m^{-2} (twice the recommended dose).

To investigate the terminal residue of flubendiamide in cabbage and soil, 20% flubendiamide WDG was applied at 0.025 and 0.05 g·m^{-2}, representing the recommended dose and twice the recommended dose, respectively. Cabbage and soil samples were collected on 0, 1, 2, 3, 5, 7, and 10 d after spraying. The depth of the soil samples was 10 cm. A minimum of 2 kg of cabbage was collected and homogenized with a blender on every sample from each plot. Soil samples (1 kg) were collected, mixed, and passed through a 1 mm sieve from each plot for analysis. All samples were stored at −20˚C prior to analysis.

2.2.2. Dispersive Solid Phase Extraction Methods

Cabbage and soil (5 g) were weighed separately in a homogenate cup, and 30 mL of acetonitrile was added. After mashing the samples by a high-speed homogenizer for 3 min, flubendiamide was added to the samples until the final concentrations of flubendiamide were 5.00, 1.00, and 0.20 μg·g^{-1}, respectively. A blank control group was also set up, and all experiments were repeated thrice. Then, 1.5 g of sodium chloride and 6 g of anhydrous magnesium sulfate were added in the samples prior to 5 min agitation in a rotary shaker at 4000 r·min^{-1}. About 2 mL of the upper mixture was collected, added to the centrifuge tubes containing $MgSO_4$ (150 mg), PSA (25 mg), and C_{18} (25 mg), and agitated for 5 min in a rotary shaker at 4000 r·min^{-1}. The sample solution (0.60 mL) was diluted to 1.0 mL of distilled water. The sample solution was filtered through a 0.22 μm membrane and subjected to LC-MS/MS chromatographic analysis in MRM mode.

2.2.3. Determinations of Flubendiamide by LC-MS/MS

The samples above were determined and analyzed by LC-MS/MS. Determination method of flubendiamide in the cabbage was described by Chen *et al*. [17]. LC analysis was performed with an Agilent 1200 HPLC system equipped with a binary pump, auto plate-sampler, column oven, and diode-array detector. Separation was performed on Agilent Eclipse Plus chromatographic columns C_{18} (4.6 mm × 150 mm (i.d.), 5 μm) at 20˚C, with mobile solvents consisting of methanol: ammonium acetate with 1% 5 mmol·L^{-1} acetic acid = 60:40 (V:V), isocratic at 1 mL·min^{-1}. Aliquots of 5 μL were injected directly to the LC-MS/MS system to test flubendiamide and quantified with external standard peak area. Mass spectra was recorded on an Agilent 6460 triple quadrupole (QQQ) mass spectrometer equipped with an ESI source. System control and data acquisition were controlled by Agilent Mass Hunter software. Detailed MS conditions were: cluster voltage: −120 V; gas temperature: 300˚C, gas flow 10 L·min^{-1}, nebulizer pressure: 15 psi, sheath gas temperature: 250˚C; sheath gas flow: 7 L·min^{-1}, capillary voltage: 4 kV, nozzle voltage: 500 V. ESI was operated in the negative ion mode in the MRM (multiple reaction monitoring).

2.2.4. Data Analysis

The kinetic equation for degradation reactions of flubendiamide accorded to first-order kinetics:

$$C_t = C_0 e^{-Kt}, \quad T_{1/2} = \frac{\ln 2}{K}$$

where $T_{1/2}$ was half life period of flubendiamide, K was the first-order rate constant, C_0 was the initial concentration and C_t was the flubendiamide concentration at time t.

3. Results and Discussion

3.1. Results of Dispersive Solid Phase Extraction Methods

The samples were allowed to settle for 30 min before using the extract and processed according to the aforementioned extraction procedure. To optimize the purification, different amounts of PSA (0, 10, 25, 50, 75, and 100 mg) and C_{18} (0, 10, 25, 50, 75, and 100 mg) were studied. The results showed no obvious improvement in the recovery of flubendiamide with increased amounts of PSA and C_{18}. When 1 mL of the cabbage extract sample was purified with adsorbents (comprising 150 mg of $MgSO_4$, 25 mg of PSA, and 25 mg of C_{18}), pesticide loss decreased and impurities were eliminated. Therefore, 25 mg of PSA, 25 mg of C_{18}, and 150 mg of $MgSO_4$ were needed for cabbage extract purification.

3.2. Fortified Recoveries of Flubendiamide from the Cabbage and the Soil

The fortified recoveries of flubendiamide from cabbage and soil were 80.70% to 90.44% and 84.37% to 94.00% as the coefficients of variation were 1.22% to 4.12% and 2.10% to 4.90%, respectively. These values were in accordance to the pesticide residue criterion at different fortification levels (**Table 1**). According to the noise signal of LC-MS/MS, the lower limit of detection of flubendiamide was 0.015 mg·kg^{-1}.

3.3. Degradation Dynamics of Flubendiamide in the Cabbage and Soil

3.3.1. Degradation Dynamics of Flubendiamide in the Cabbage

Compared with the LC-MS/MS figure of flubendiamide standard, the LC-MS/MS figure of the cabbage sample exhibited intense ion fragmentation at *m/z* 254, 272, 274, and 214 in the MRM negative mode. The residue in the cabbage sample was proven to be flubendiamide (**Figure 1** and **Figure 2**). The results showed that the degradation dynamic equations of flubendiamide in the cabbage were based on the first-order reaction dynamic equations, and the half-lives of the degradation of flubendiamide were 3.51 d to 3.96 d and 3.43 d to 3.87 d in the cabbage of Yangzhou and Jingzhou, respectively (**Table 2**).

Table 1. The recoveries of flubendiamide from the cabbage and the soil.

Sample	Fortification Levels (mg·kg^{-1})	Average Recoveries (%)	Standard Deviation	Coefficient of Variance (%)
Cabbage	5.0	90.44	0.0551	1.22
	1.0	89.83	0.0148	1.64
	0.2	80.70	0.0066	4.12
Soil	5.0	94.00	0.0988	2.10
	1.0	88.30	0.0260	2.95
	0.2	84.37	0.0083	4.90

Note: Mean value of three replicates.

Table 2. Degradation dynamics equation of flubendiamide in the cabbage.

Time (year)	Sample Location	Degradation Dynamics Equation	Correlation Coefficient	Half-Life (d)
2011	Yangzhou	$C_t = 0.1865e^{-0.1750t}$	0.9812	3.96
	Jingzhou	$C_t = 0.1937e^{-0.1791t}$	0.9844	3.87
2012	Yangzhou	$C_t = 0.2007e^{-0.1977t}$	0.9865	3.51
	Jingzhou	$C_t = 0.2106e^{-0.2021t}$	0.9783	3.43

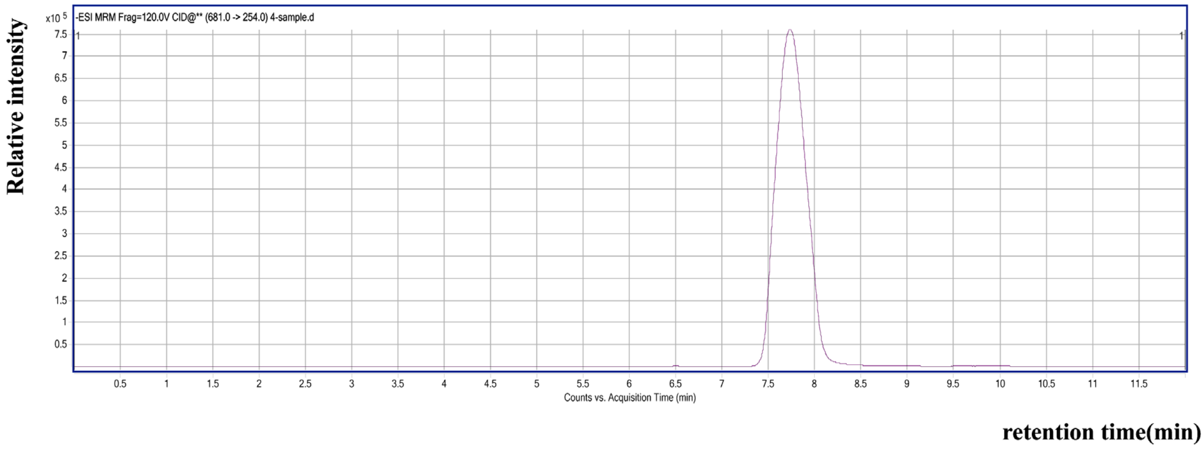


Figure 1. Selected ionization chromatography of residual flubendiamide in the cabbage sample by MRM mode (*m/z* 254).

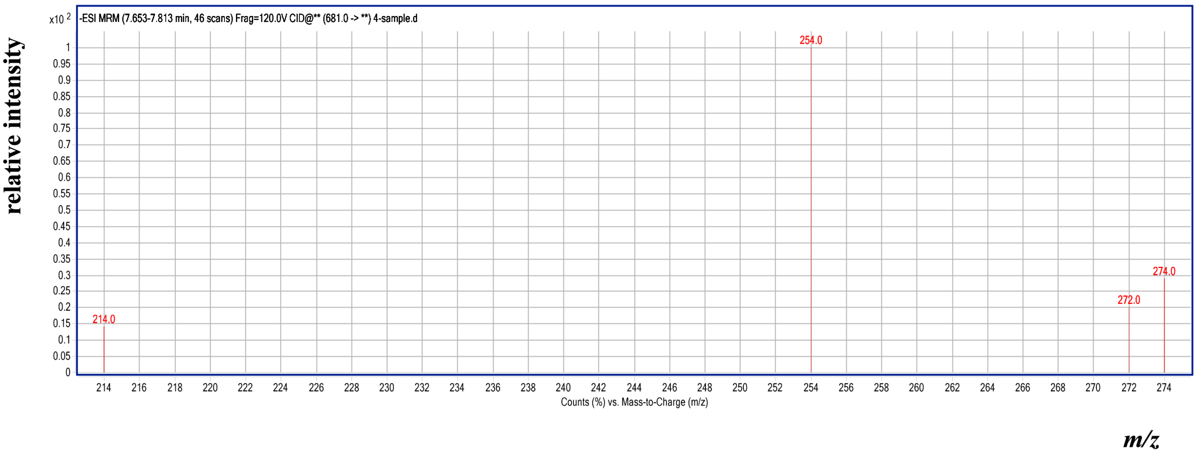


Figure 2. MS-MS spectrum of residual flubendiamide in the cabbage sample by LC-MS/MS.

3.3.2. Degradation Dynamics of Flubendiamide in the Soil

Compared with the LC-MS/MS figure of flubendiamide standard, the LC-MS/MS figure of the soil sample exhibited intense ion fragmentation at *m/z* 254, 272, 274, and 214 in the MRM negative mode. The residue in the soil sample was proven to be flubendiamide (**Figure 3** and **Figure 4**). The results showed that the degradation dynamic equations of flubendiamide in the soil were based on the first-order reaction dynamic equations, and the half-lives of the degradation of flubendiamide were 4.42 d to 5.13 d and 4.37 d to 4.99 d in the soil of Yangzhou and Jingzhou, respectively (**Table 3**).

3.4. Terminal Residue of Flubendiamide in the Cabbage and Soil

3.4.1. Terminal Residual of Flubendiamide in the Cabbage

The terminal residue of flubendiamide in the cabbage of Yangzhou was not detected, but 0.0247 mg·kg^{-1} to 0.0393 mg·kg^{-1} was detected when 20% flubendiamide WDG was applied at concentrations of 0.0250 and 0.050 g·m^{-2}. The terminal residue of flubendiamide in Jingzhou was not detected, but 0.0225 mg·kg^{-1} to 0.0273 mg·kg^{-1} was detected when 20% flubendiamide WDG was applied at concentrations of 0.0250 and 0.050 g·m^{-2} (**Table 4**). The residue levels markedly lowered the MRL standard in the cabbage [MRL = 3 mg·kg^{-1} (Japan); MRL = 11 mg·kg^{-1} (USA); MRL = 4 mg·kg^{-1} (Australia)]. Harvest intervals were suggested to ensure that the cabbage was sufficiently safe for consumption at the recommended dose.

3.4.2. Terminal Residual of Flubendiamide in the Soil

The terminal residue of flubendiamide in the soil of Yangzhou was not detected, but 0.0691 mg·kg^{-1} to 0.0762 mg·kg^{-1} was detected when 20% flubendiamide WDG was applied at concentrations of 0.0250 and 0.050 g·m^{-2}. The terminal residue of flubendiamide in the soil of Jingzhou was not detected, but 0.0475 mg·kg^{-1} to 0.0589 mg·kg^{-1} was detected when 20% flubendiamide WDG was applied at concentrations of 0.0250 and 0.050 g·m^{-2} (**Table 5**).

A number of techniques exist for the pretreatment of samples, including various solvent extraction methods, solid phase extraction or gel permeation chromatography clean up, followed by gas chromatography analysis using a variety of detectors [18]-[20]. Given that all these procedures are complex, require the use of toxic solvents, and are time consuming, an alternative technique called dispersive solid phase extraction was applied. This approach has wide analytical scope and high degree of selectivity and sensitivity. The method solves various problems, including low injection dosage, long analysis time and extensive use of poisonous solvents. Compared with other techniques, it has the following strong points: 1) As a multi-residue analytical method, it can reduce interferences from the sample matrix, such as chlorophyll, grease and moisture; 2) It has good stability and high recovery, and the recovery of numerous polar and volatile substances is over 85%; 3) The analysis time is short as determination can be done in 10 min to 20 min; 4) It uses a minimum amount of solvent, is inexpensive, and results in less pollution [21]-[23]. Dispersive solid phase extraction combined with LC- MS/MS method was applied in this study, according to the noise signal of LC-MS/MS, the lower limit of detection of

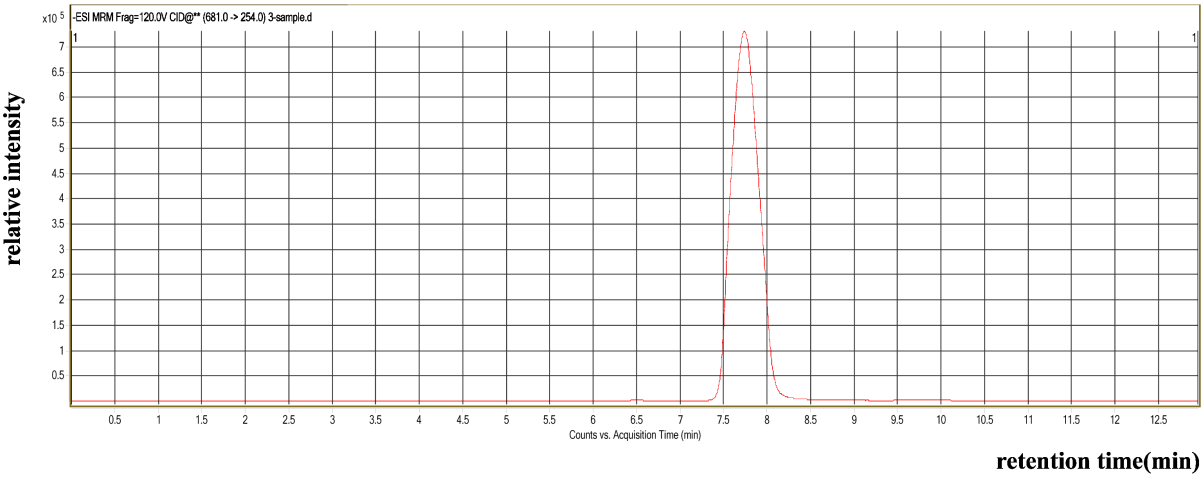


Figure 3. Selected ionization chromatography of residue flubendiamide in the soil sample by MRM mode (*m/z* 254).

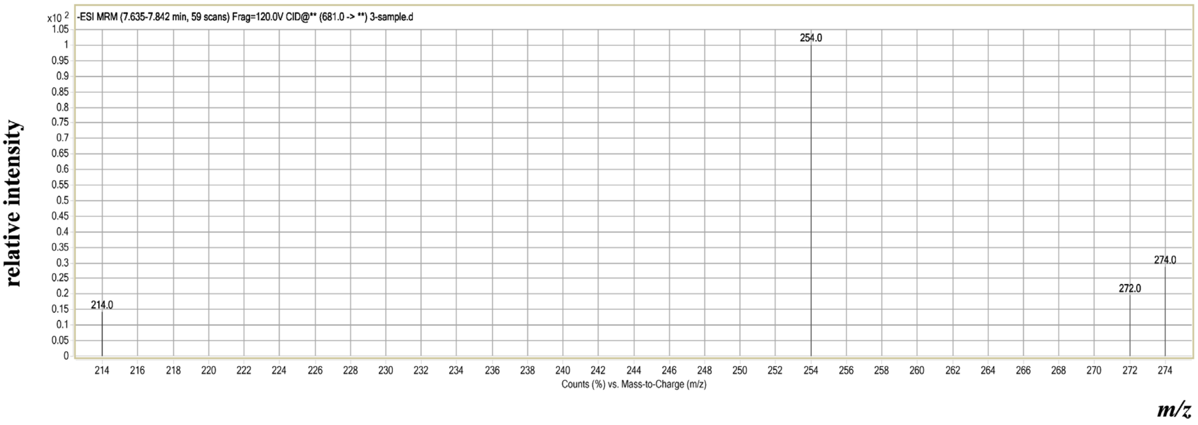


Figure 4. MS-MS spectrum of residue flubendiamide in the soil sample by LC-MS/MS.

Table 3. Degradation dynamics equation of flubendiamide in the soil.

Time (year)	Sample Location	Degradation Dynamics Equation	Correlation Coefficient	Half-Life (d)
2011	Yangzhou	$C_t = 1.8131e^{-0.1352t}$	0.9770	5.13
	Jingzhou	$C_t = 1.9875e^{-0.1389t}$	0.9804	4.99
2012	Yangzhou	$C_t = 2.0251e^{-0.1569t}$	0.9797	4.42
	Jingzhou	$C_t = 1.8051e^{-0.1586t}$	0.9863	4.37

Table 4. Terminal residual of flubendiamide in the cabbage.

Application Dose ($\text{g}\cdot\text{m}^{-2}$)	Terminal Residual of Yangzhou ($\text{mg}\cdot\text{kg}^{-1}$)		Terminal Residual of Jingzhou ($\text{mg}\cdot\text{kg}^{-1}$)	
	2011	2012	2011	2012
0.025	No	No	No	No
0.050	0.0393	0.0247	0.0273	0.0225
CK	No	No	No	No

Note: Mean of three replicates. "No" means no flubendiamide was detected.

flubendiamide was 0.015 $\text{mg}\cdot\text{kg}^{-1}$, which could provide high-quality results for degradation dynamics and residue analysis of flubendiamide in cabbage and soil qualitatively and quantitatively. The method is rapid, simple, highly sensitive, and can meet the requirements for analysis of active ingredients and residues.

Table 5. Terminal residual of flubendiamide in the soil.

Application Dose ($g \cdot m^{-2}$)	Terminal Residual of Yangzhou ($mg \cdot kg^{-1}$)		Terminal Residual of Jingzhou ($mg \cdot kg^{-1}$)	
	2011	2012	2011	2012
0.025	No	No	No	No
0.050	0.0762	0.0691	0.0589	0.0475
CK	No	No	No	No

Note: Mean of three replicates. "No" means no flubendiamide was detected.

4. Conclusion

In conclusion, the recoveries of flubendiamide from the cabbage and the soil were 80.70% - 90.44% and 84.37% - 94.00%, respectively. Degradation dynamic equations of flubendiamide in the cabbage and soil were based on the first-order reaction dynamic equations. The half-life of the degradation of flubendiamide was 3.51 - 3.96 d and 3.43 - 3.87 d in the cabbage of Yangzhou and Jingzhou respectively. The half-life of the degradation of flubendiamide was 4.42 - 5.13 d and 4.37 - 4.99 d in the soil of Yangzhou and Jingzhou, respectively. The terminal residue of flubendiamide in the cabbage of Yangzhou and Jingzhou was 0.0247 - 0.0393 $mg \cdot kg^{-1}$ and 0.0225 - 0.0273 $mg \cdot kg^{-1}$ when 20% flubendiamide WDG was applied at the using dose of 0.050 $g \cdot m^{-2}$. Flubendiamide should be safe if it was applied in the cabbage field at the recommended dose.

Acknowledgements

The authors would like to thank the Natural Science Foundation of Jiangsu Province (BK20130443), Social Development Science and Technology Projects of Yangzhou City, China (2012110) and Natural Science Foundation of the Jiangsu Higher Education Institutions of China (13KJB210010) for providing financial support.

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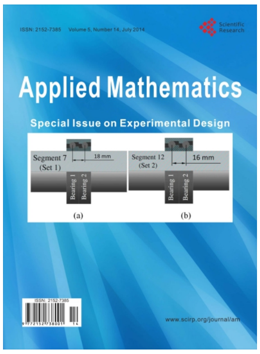
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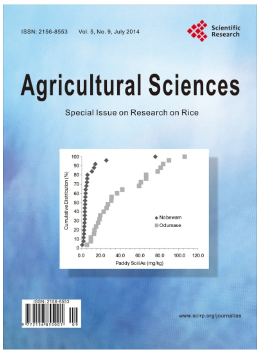
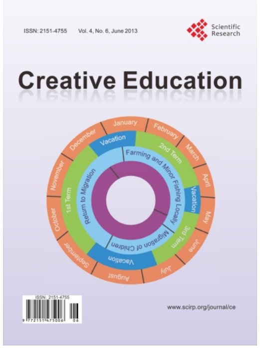

Agricultural Sciences
Special Issue on Research on Rice

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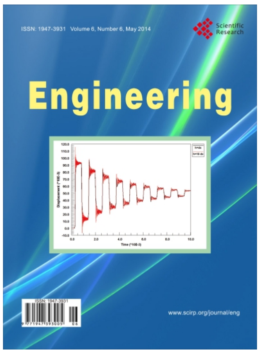
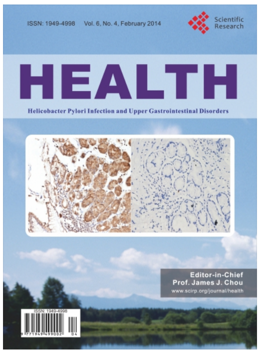
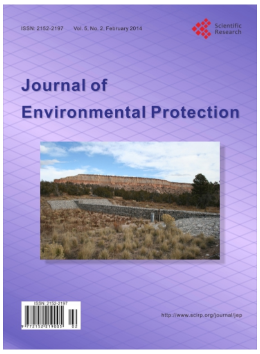

Engineering

Food and Nutrition Sciences

HEALTH
Helicobacter Pylori Infection and Upper Gastrointestinal Disorders

Journal of Computer and Communications

Journal of Cancer Therapy
Special Issue on Bone Tumor Research

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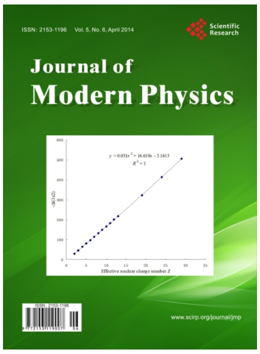
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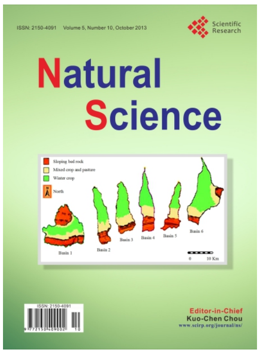
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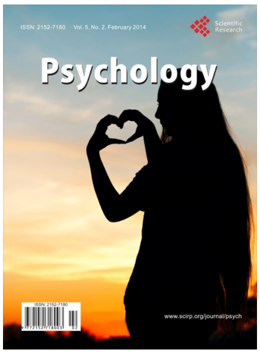
Psychology