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Research Article

Development of ultra HPLC analytical method for pymetrozine residues in rice

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Abstract

Ultra HPLC-based analytical method analytical method was developed for the analysis of pymetrozine residues in different rice matrices. Chromatography separation was carried out in XR-ODS II: 150mm X 2.0mm, 5µm column in the mobile phase of water and methanol (60:40), and the resultant chromatograms were detected in a Photodiode Array (PDA). The retention time for pymetrozine neat standard and also matrix match standards was arrived at 4.00 min ± 0.5 min. The pymetrozine at the concentrations of 0.01, 0.05, 0.10, 0.25, 0.50, 0.75, and 1.00 µg/ml and the calibration curve of $y = 2E^{+07}x + 19188$ obtained with a goodness of fit (R^2) 0.995. The samples were extracted using the AOAC QuEChERS method with slight modifications. The harvest time residues for the different rice matrices were below the tolerance limit of pymetrozine. The residues of pymetrozine at 7, 15, and 30 days after the rice harvest in paddy seed, single-polished, double-polished rice, bran, and dehulled rice were not detected. From the developed analytical method, the residues of pymetrozine can be detected in different rice matrices.

Introduction

Pymetrozine,4,5-dihydro-6-methyl-4-[(3-pyridylmethylene)-amino]-1,2,4-triazin-3(2H)-one (Figure 1), is a novel insecticide with effects on neuroregulation or nerve-muscle interaction against sucking insects by preventing these ones from inserting their stylus into the plant tissue, reduction in feeding and alteration in probing behaviors characterized by the prolonged non-probing, penetration initiation and extracellular activity and shortened salivation and phloem-related activities [1-3]. Pymetrozine has been widely used in rice against plant hoppers [4] as a substitution for highly toxic organophosphate pesticides and vegetables [5]. Though pymetrozine is registered for use in various crops and vegetables because of its broad-spectrum activity, the EPA has considered that pymetrozine is a "likely" human carcinogen and is slight to moderately toxic to aquatic invertebrates [6]. Therefore, pymetrozine residues may lead to potential health injury. Several analytical methods have been developed for qualitative and quantitative analysis of pymetrozine residues in different matrices, such as Differential Pulse Polarographic method (DPP), Liquid Chromatography (LC) with UV/vis, Diode Array Detection (DAD), and Mass Spectrometry (MS); however, the fast and efficient method is LC with mass spectrometry (MS) [7-11].

The studies pertaining to harvest time residues of pymetrozine in different rice matrices are limited. A simple, sensitive, and selective method with Ultra High-Performance Liquid Chromatography (UHPLC) for quantification of pymetrozine in different rice matrices *viz.*, rice grain, single and double polished rice, rice bran was developed to study the dissipation behavior and final residue of pymetrozine in rice. The present investigations were carried out at the Pesticide Residue Testing Laboratory, RARS, Tirupati during 2022-2023.

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Materials and methods

Sample collection and processing

The rice crop was grown in a bulk plot of 50m² as per the standard agronomic practices of ANGRAU during post rainy season of 2022-2023 and kept unsprayed. One week before harvest pymetrozine 50WP was sprayed. Approximately two kg samples were collected at the time of harvest, 7, 15, and 30 days after harvest. Samples were processed at ARS, Nellore for dehull rice, single polished rice, double-polished rice, and bran from single and double-polished rice and brought to pesticide residue laboratory, IFT, RARS, Tirupati.

Chemicals and reagents

Technical grade standard of pymetrozine (purity:99.8%, make: Sigma-Aldrich), HPLC grade methanol, n-Hexane, and water Merck make, QuEChERS extraction salts like sodium sulphate anhydrous, sodium acetate, magnesium sulfate anhydrous were purchased from local vendors. Primary Secondary Amine, also obtained Sigma-Aldrich make. The formulation of pymetrozine (50WG) for field experiments was purchased from a local agricultural pesticide market.

Instrument

Ultra High-Performance Liquid Chromatography (UHPLC) Shimadzu make, with SPD-M20A, PDA detector. Deuterium (D2) and tungsten (W) lamps, which can read a wavelength of 190 to 800 nm were used for analysis.

Instrumentation

Chromatography was performed in a Shimadzu Ultra HPLC system equipped with a Photo Diode Array detector. Chromatographic separation was achieved in column (XR-ODS II: 150mm X 2.0mm), and the column oven temperature was set at 32.2°c. The injection volume was 10µl, the pump was LC-30AD which was set at a low-pressure gradient equipped with a flow rate of 0.8µl/min. The mobile phase set with Pump A: Water (60%), Pump B: methanol (40%) at a Pump Flow of 0.8ml/min. The Photodiode array (PDA) detector (SPD-M20A) is equipped with a D2 (Deuterium) lamp. The conditions pertaining to LC conditions are presented in Table 1.

Preparation of standard solution

Approximately 10 ± 0.01mg of pymetrozine standard was weighed into a 10 ml standard flask and diluted with methanol to prepare a stock solution of 1000 µg/ml. The standard solution was further diluted to 100 µg/ml, and 10 µg/ml.

The standard solution was diluted into different concentration gradients. Linear concentrations of 0.01, 0.05, 0.10, 0.25, 0.50, 0.75 and 1.00 µg/ml were prepared for drawing the standard curve, the quality concentration was horizontal coordinates and the peak area was vertical coordinates, so as to obtain a series of linear regression equations in different rice matrices were determined, the correlation coefficient was calculated for the goodness of the methodology.

Table 1: Details of the UHPLC -PDA Operational Parameters.

S. No	Parameter	Conditions
1	Liquid chromatography	Shimadzu
2	Pump	LC-30AD quaternary pump system
3	Pump mode	Low-pressure gradient
4	Column	XR-ODS II: 150mm X 2.0mm, 5µm
5	Oven Temperature	32.2 °C
6	Injection volume & Interval	10 µl in 10 min
7	Total flow	0.8 ml/min
8	Mobile phase	Pump A- Water (60%) Pump B - Methanol (40%)
9	Wavelength	272 nm
10	Detector	Photodiode array (PDA)
11	Detector mode	SPD-M30A
12	Lamp	D2 (Deuterium)
13	Total run time	10.0 min
14	Retention time	3.80 min

Method development and validation

The suitability of the instrument for analyzing the pymetrozine was tested by injecting the pure standard at various concentrations and methanol as blank in the methanol-water solvent system. The following parameters were validated in method development (based on the external standard method).

1. Retention time
2. Linearity and range
3. Limit of Detection (LOD) and Limit of Quantification (LOQ)
4. Accuracy and Precision
5. Recovery

Retention time: Retention time is the primary means for chromatographic peak identification. Predicting chromatographic retention times of pesticides has become more and more important for suspect and non-target screening. Pymetrozine was analyzed in reverse phase XR-ODS IIcolumn measured about 150mm×2.0mm, 5µm for separation. The retention time for pymetrozine was analyzed in different combinations of methanol and HPLC water. Pymetrozine standard was run in the solvent system of 40:60 methanol and water in comparison with methanol blanks. The retention time for other concentrations of pymetrozine (0.01, 0.05, 0.1, 0.25, 0.50, 0.75, and 1.00 µg/ml was also studied in replicates).

Selectivity of the method: The selectivity of the method was evaluated by injecting extracts of the blank rice matrices and methanol followed by a comparison of the chromatograms obtained with extracts of the rice matrices fortified with the analytes. The signal absence in the chromatograms for the blank samples in the retention times of all the concentrations of analytes studied confirmed the selectivity of the method.

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Linearity, range, and calibration: It is very essential to calibrate the instrument to obtain accurate results. The linearity of an analytical procedure is its ability to obtain test results within a given range that is directly proportional to the concentration of an analyte in the sample. It is evaluated by linear regression analysis of the plot of signals as a function of analyte concentration with a minimum of five linear concentrations. The correlation coefficient and slope of the regression line should be satisfactory.

A stock solution of pymetrozine (1000 µg/ml) pipetted out, and linear concentrations of 0.01, 0.05, 0.1, 0.25, 0.50, 0.75, and 1.00ppm were prepared and injected into the Ultra HPLC instrument in three replications. The peak areas were measured and tabulated. The detector response in terms of peak areas by standard solutions of pymetrozine (0.1 to 1 µg /ml in mobile phase) was measured.

Matrix-match calibration: Rice matrices a sample without pymetrozine was extracted in the same procedure adopted for other samples. After extraction, the pymetrozine was added at different concentrations *i.e.,* 0.01, 0.05, 0.10, 0.25, 0.50, 0.75, and 1.00 µg/ml for constructing calibration and goodness of fit. Linear calibration graphs were prepared and goodness of fit was studied.

Limit of Detection (LOD) and Limit of Quantification (LOQ): The limit of detection for pymetrozine was considered to be the concentration that produced a S/N ratio of 3:1 and the LOQ was defined on an S/N ratio of 10:1. These values were estimated from the chromatogram corresponding to the lowest concentration that could produce a response of 3:1 from different concentrations injected. Precision was arrived by injecting the pymetrozine 0.50ppm concentration in six replicates.

Trueness and precision

The trueness was determined from the recovery assay results of samples spiked with the analytes at 0.05 and 0.10 mg kg^{-1} (n = 3 replicates per level) along with blank samples. Repeatability, expressed as Relative Standard Deviation (RSD), was evaluated through the data from replicated samples analyzed on the same day for each level. The intermediate precision, expressed as Relative Standard Deviation (RSD), was evaluated through the replicated data on the three different days for each level.

Recovery study: The reliability of the analytical method chosen and the efficiency of extraction and clean-up procedures were verified by carrying out recovery experiments in the laboratory. To 10 g rice samples like paddy seed, dehulled rice, single polished, double-polished rice, single polished bran, double-polished bran 0.05, 0.10, and 0.50 mg kg–1 of analytical pymetrozine solutions were added, mixed thoroughly, and kept for 30 minutes. The pymetrozine residues in the fortified samples were then extracted, cleaned up, and estimated by the same method as per samples. A sample without insecticide fortification and a blank sample (methanol) were maintained as untreated control for comparison.

Extraction of pymetrozine from rice matrices

Extraction: The samples were extracted for pymetrozine as per the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) method with slight modifications. The samples were course ground and weighed approximately 10 ± 0.1g of rice sample (dehulled rice, single polished, double-polished rice, and bran, paddy seed) into a 50ml centrifuge tubes and added 10ml of ice-cold distilled water and vortexed. The known concentration of pymetrozine at 0.05, 0.10, and 0.50 µg/ml was added to each sample on a weight/volume basis. The samples were vortexed for mixing of the pymetrozine in the matrix and added 20ml methanol, capped well, and shaken for thorough mixing.

The samples were homogenized using silent crusher at 14000 to 15000rpm for 2-3 min, added 3 ± 0.1g Sodium Chloride and vortexed for a minute, and centrifuged the contents at 2500-3000rpm for min to separate the organic layer and approximately 12 ml of supernatant was transferred to a test tube and added 5.0±0.1g anhydrous sodium sulfate to remove the moisture content.

Dispersive solid phase clean-up

Weighed 0.20 ± 0.01g PSA sorbent and 0.60 ± 0.01g anhydrous magnesium sulfate in a 15ml centrifuge tube and 9ml of upper organic layer from anhydrous sodium sulfate treated test tube was transferred. The contents were vortexed and centrifuged at 2500-3000rpm for 5 min and the upper 1ml layer was filtered through a PTFE (0.22 µm) syringe filter to an HPLC vial for analysis.

Recovery studies

The rice matrices *viz.*, rice grain with husk, dehulled rice, single polished rice, double-polished rice, and rice bran from single and double polished rice were spiked with pymetrozine at various concentrations 0.01, 0.05 and 0.10ppm. The extraction and clean-up were done in the same manner the sample extraction was done.

Sample analysis and calculation of residues from rice matrices

Residue was calculated by the following formula

$$\text{Residue}(\mu g / g) = \frac{\text{Area of sample}}{\text{Area of standard}} \times \frac{\text{Conc. of Standard in } \mu g / ml}{\text{Weight of sample}(g)} \times \text{Dilution factor}$$

After analysis, pymetrozine residues have been calculated and expressed in mg kg^{-1}.

Results

Retention time

The retention time for pymetrozine was analyzed in different combinations of methanol and HPLC water. The retention time of 3.80 min for 1.0ppm pymetrozine was obtained in a solvent system of 60: 40 water and methanol. The retention time for other concentrations of pymetrozine *i.e.,* 0.01, 0.05, 0.1, 0.25,

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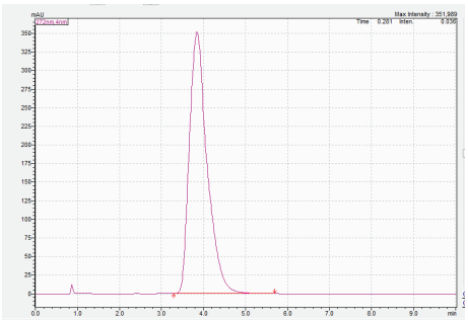

Figure 1a: Pymetrozine @ 1.0ppm.

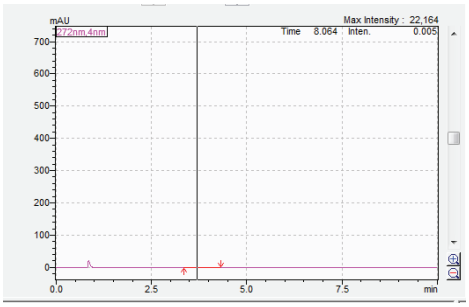

Figure 1b: Solvent Blank.

0.50, 0.75ppm also at 3.80 ± 0.5 min, whereas, the peak at the corresponding retention time was not observed in solvent blank (Figure 1a,b).

Linearity and range

Seven concentrations of pymetrozine *i.e.*, 0.01, 0.05, 0.1, 0.25, 0.50, 0.75, and 1.00 ppm were prepared and injected into the Ultra HPLC instrument in three replications. The peak areas were measured and tabulated. The detector response in terms of peak areas by standard solutions of pymetrozine (0.01 to 1 µg /ml in mobile phase) was measured. There was a good correlation between the concentration of pymetrozine injected and the resultant chromatogram obtained. The regression equation obtained was $y = 2E^{+07}x + 191888$ with goodness of fit (R^2) of 0.996 (Table 2 and Figure 2). Similarly, the matrix match calibration for rice matrices also studied for the linearity and found the R^2 of more than 98 percent for the six rice matrices.

Limit of detection (LOD) and Limit of Quantification (LOQ)

There was a progressive increase in the chromatogram area with increasing concentrations for pymetrozine standard. A signal-noise ratio of 3 is considered for the limit of detection (LOD) and 10 for limit of quantification. For pymetrozine, an S/N of 3 was observed at 0.01 ppm and 0.10 ppm.

Matrix match calibration of pymetrozine in different rice matrix

Calibration studies pertaining to pymetrozine neat standard and matrix match calibrations are presented in the Table 2. The regression equation for neat pymetrozine standard at the concentrations of 0.01, 0.05, 0.10, 0.25, 0.50, 0.75, 0.10 is $y = 2E^{+07}x + 191888$ with $R^2 = 0.996$. Similarly, matrix match calibration of various rice matrices was calculated for the suitability of the method for pymetrozine analysis in rice (Table 2 and Figures 3-8).

Recovery

The recovery studies for pymetrozine were studied at 0.05ppm spiked concentration. The recovery of pymetrozine in different rice matrices indicated that a recovery in the range of 70-120 percent was observed. In dehulled rice, single polished rice, and double polished rice it was 99.99, 101.36, and 96.73 percent, respectively. In single-polished bran, and double-polished bran it was 77.72 and 106.90 percent and in rice grain, it was 111.93 percent (Table 3).

Table 2: Matrix match calibration of pymetrozine in different rice matrix.

Particulars	Regression equation	R^2
Pymetrozine standard	$y = 2E^{+07}x + 191888$	$R^2 = 0.99$
Double-polished rice matrix	$y = 1E^{+06}x + 22238$	$R^2 = 0.99$
Single-polished rice matrix	$y = 1E^{+06}x - 1158.7$	$R^2 = 0.99$
Dehulled rice	$y = 2E^{+06}x - 18647$	$R^2 = 0.99$
Rice husk matrix	$y = 818703x + 14899$	$R^2 = 0.99$
Double-polished bran	$y = 2E^{+06}x - 7148.7$	$R^2 = 1.00$
Single-polished bran	$y = 2E^{+06}x + 20959$	$R^2 = 0.99$

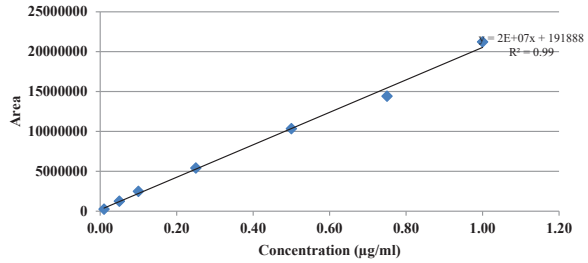

Figure 2: Calibration curve of Pymetrozine neat standard.

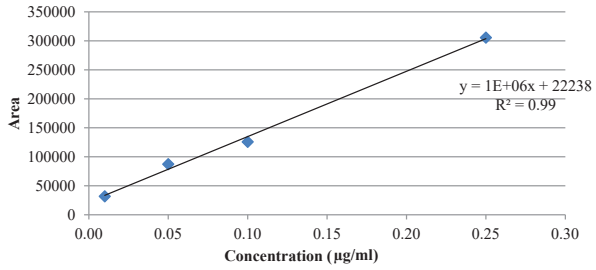

Figure 3: Calibration curve of pymetrozine spiked in double polished rice matrix.

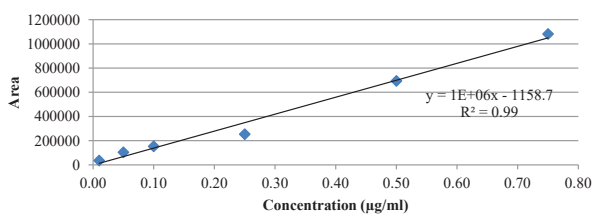

Figure 4: Calibration curve of Pymetrozine spiked in single polished rice matrix.

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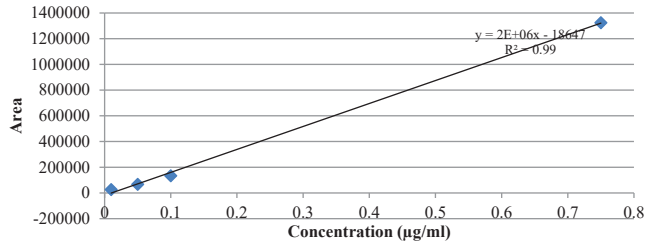

Figure 5: Calibration curve of pymetrozine spiked in dehulled rice.

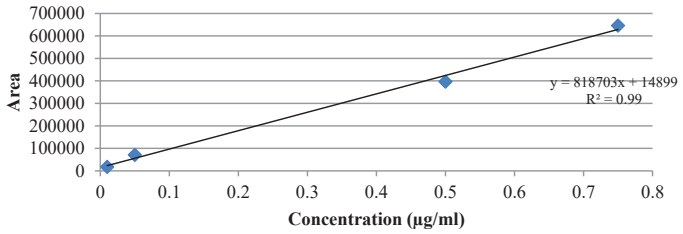

Figure 6: Calibration curve of pymetrozine spiked in rice husk matrix.

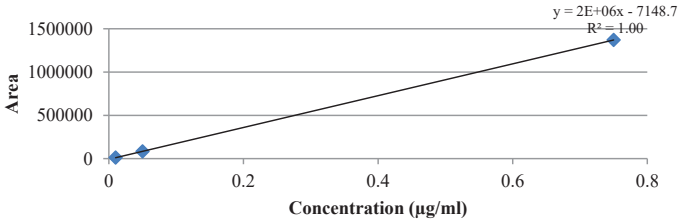

Figure 7: Calibration curve of pymetrozine spiked in double polished bran matrix.

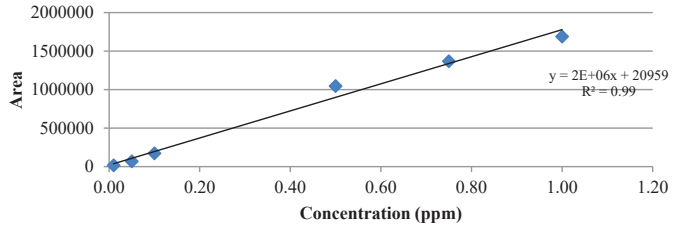

Figure 8: Calibration curve of pymetrozine spiked in single polished bran matrix.

Table 3: Recovery of pymetrozine in different rice matrices.

Commodity		Recovery @ 0.05ppm				
		R1	R2	R3	Mean	± SD
Dehusked rice	ppm	0.060	0.047	0.044	0.050	0.009
	%	119.55	93.31	87.13	99.99	17.21
Single Polished Rice	ppm	0.058	0.047	0.047	0.051	0.006
	%	116.03	93.87	94.18	101.36	12.71
Double Polished Rice	ppm	0.050	0.050	0.045	0.048	0.003
	%	100.09	100.31	89.80	96.73	6.00
Single Polished Bran	ppm	0.086	0.072	0.075	0.078	0.008
	%	86.37	71.76	75.04	77.72	7.67
Double Polished Bran	ppm	0.051	0.049	0.060	0.053	0.005
	%	102.42	98.99	119.29	106.90	10.87
Paddy	ppm	0.056	0.056	0.056	0.056	0.000
	%	112.34	111.41	112.05	111.93	0.47

Residues of pymetrozine

The developed Ultra HPLC method after validation was applied to test the residues of pymetrozine in various rice matrices. The residues of pymetrozine at harvest of paddy crop in different matrices, including rice grain with husk, dehulled rice, single polished rice, double-polished rice, and rice bran, were below the tolerance limit at harvest, and they were below detectable levels in all the test matrices at 7, 15, and 30 days after storage.

Discussion

In the present study, a method was developed for residue analysis of pymetrozine in Ultra HPLC in a solvent system of water: methanol (60:40) at a flow rate of 0.8ml. Being a polar pesticide, Ultra HPLC was selected for pymetrozine. The method had good linearity (R^2 = 0.99) for pure pymetrozine standard and also rice matrices fortified with pymetrozine. The RSD for pure pymetrozine concentrations was 0.04 to 1.97 and it was less than 20 percent in different sample matrices of rice at the spiked concentrations. The QuEChERS method of extraction is suitable and results in good recoveries of 77.82 to 111.93 percent in various rice matrices.

Similarly, an HPLC method for analyzing the residues and persistence of pymetrozine green tobacco leaves. The extraction was done in acetonitrile water and clean-up was done using SPE cartridges. Average recoveries ranged from 97-99% with RSDs below 2.1%. The limit of detection was 0.05 µg/ml [12].

Gong, et al. [13] also developed a method for residue analysis of pymetrozine and its metabolites in Chinese kale in a simple determination method using liquid chromatography with tandem mass spectrometry. The developed method had good linearity (R^2 > 0.99), accuracy (recoveries of 73.2 – 94.1%), and precision (relative standard deviation of 2.5–9.8%). Field results showed that half-lives of pymetrozine were 3.0–4.1 d in Chinese kale, and terminal residue concentrations were all below the United States Environmental Protection Agency's maximum residue limit (250 µg/kg) at harvest.

Muyesaier Tudi, et al. [14] developed a liquid chromatography method for the residue analysis of pymetrozine in rice soils, and rice fields. The results of the method performance showed that the recovery ranges for both two soils and water were between 70% and 120%, and the RSD was lower than 20% in this study, being within the accepted level for residue determination settled by Document No. Nº SANTE/11312/2021 (European Commission, 2011). The analytic methods for both the red soil in Hunan and the loamy soil in Guangxi meet the requirement. The Limit of Detection (LOD) is the lowest concentration of analyte detectable by an analytical method and it was expressed in concentration units. The LOD calculated as a sample concentration was 0.1 µg/kg in soil and 0.1 µg/L in water, respectively.

The present studies were comparable with Yuting Chen, et al. [15] who developed HPLC-MS / MS method based method for pymetrozine residue analysis in *Solanum tuberosum* L and *Chrysanthemum morifolium* (Ramat). A dietary risk assessment

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of pymetrozine in *S. tuberosum* and *C. morifolium* samples was performed using these data. The detection rates of pymetrozine in *S. tuberosum* and *C. morifolium* samples were 92.31% and 98.17%, respectively, with residues not more than 0.036 and 0.024 mg/kg, respectively.

Conclusion

In the present study, an ultra HPLC-PDA-based analytical method was developed for analyzing the residues of pymetrozine residues in different rice matrices. Chromatographic separation was done in XR-ODS II: 150 mm X 2.0 mm, 5µm, and chromatograms were detected in a photodiode array detector. The samples were extracted using the QuEChERS method with slight modifications. The retention time for pymetrozine was observed at 4.00 min ± 0.5 min. The calibration curves of pymetrozine pure standard as well matrix matches with various rice commodities observed were linear for the dose vs. area with a goodness fit of 0.99. The results of the method performance showed that the recovery ranges for various rice matrices were between 70% and 120%, and the RSD was lower than 20% in this study, being within the accepted level for residue determination settled by Document No. SANTE/2019/12682. The developed HPLC-PDA-based analytical method is highly suitable for analyzing pymetrozine residues in different rice matrices.

References

1. Yu J, Zhong Y, Dai C, Hou M. Sublethal concentrations of pymetrozine reduce Sogatella furcifera transmission of Southern rice black-streaked dwarf virus. Pest Manag Sci. 2024 Feb;80(2):797-804. doi: 10.1002/ps.7815. Epub 2023 Oct 16. PMID: 37794233.

2. Li C, Yang T, Huangfu W, Wu Y. Residues and dynamics of pymetrozine in rice field ecosystem. Chemosphere. 2011 Feb;82(6):901-4. doi: 10.1016/j.chemosphere.2010.10.053. Epub 2010 Nov 11. PMID: 21074245.

3. Wang W, Cho YJ, Song JW, Kim YJ, Seo JS, Kim JH. Residue Behavior of Methoxyfenozide and Pymetrozine in Chinese Cabbage and Their Health Risk Assessment. Foods. 2022 Sep 26;11(19):2995. doi: 10.3390/foods11192995. PMID: 36230071; PMCID: PMC9563777.

4. Xu WM, Zhang M, Wei K, Chen Y, Liu Q, Xue W, Jin LH, He M, Chen Z, Zeng S. Development and evaluation of pymetrozine controlled-release formulation to control paddy planthopper. 8(40): 22687–22693. doi: 10.1039/c8ra03516d.

5. Choudhary S, Choudhary S, Sharma M, Kumawat KC, Meena RK. Bioefficacy of novel insecticide molecules against sucking insect pests of Indian bean. The Pharma Innovation Journal. 2022; SP-11(5): 833-838.

6. Cho Y, Jeon HJ, Kim K, Kim C, Lee SE. Developmental toxicity of a pymetrozine photo-metabolite, 3-pyridinecarboxaldehyde, in zebrafish (Danio rerio) embryos: Abnormal cardiac development and occurrence of heart dysfunction via differential expression of heart formation-related genes. Ecotoxicol Environ Saf. 2023 Mar 15;253:114654. doi: 10.1016/j.ecoenv.2023.114654. Epub 2023 Feb 15. PMID: 36801540.

7. Guan W, Li Z, Zhang H, Hong H, Rebeyev N, Ye Y, Ma Y. Amine modified graphene as reversed-dispersive solid phase extraction materials combined with liquid chromatography-tandem mass spectrometry for pesticide multi-residue analysis in oil crops. J Chromatogr A. 2013 Apr 19;1286:1-8. doi: 10.1016/j.chroma.2013.02.043. Epub 2013 Feb 20. PMID: 23489497.

8. Abd Al-Rahman SH, Almaz MM, Ahmed NS. Dissipation of fungicides, insecticides, and acaricide in tomato using HPLC-DAD and QuEChERS Methodology. Food Analytical Methods. 2012; 5: 564–570.

9. Lehotay SJ, Son KA, Kwon H, Koesukwiwat U, Fu W, Mastovska K, Hoh E, Leepipatpiboon N. Comparison of QuEChERS sample preparation methods for the analysis of pesticide residues in fruits and vegetables. J Chromatogr A. 2010 Apr 16;1217(16):2548-60. doi: 10.1016/j.chroma.2010.01.044. Epub 2010 Jan 22. PMID: 20144460.

10. Mercan H, Yilmaz E, Inam R. Determination of insecticide pymetrozine by differential pulse polarography/application to lake water and orange juice. J Hazard Mater. 2007 Mar 22;141(3):700-6. doi: 10.1016/j.jhazmat.2006.07.029. Epub 2006 Jul 21. PMID: 16949742.

11. Lee SW, Choi JH, Cho SK, Yu HA, Abd El-Aty AM, Shim JH. Development of a new QuEChERS method based on dry ice for the determination of 168 pesticides in paprika using tandem mass spectrometry. J Chromatogr A. 2011 Jul 15;1218(28):4366-77. doi: 10.1016/j.chroma.2011.05.021. Epub 2011 May 14. PMID: 21632058.

12. Juju S, Xinsheng C, Lan H, Xu C. Solid Phase Extraction and HPLC analysis of pymetrozine residues in green tobacco leaves. Asian Journal of Chemistry. 2010; 22(4): 2774-2782.

13. Gong J, Zheng K, Yang G, Zhao S, Zhang K, Hu D. Determination, residue analysis, risk assessment and processing factor of pymetrozine and its metabolites in Chinese kale under field conditions. Food Addit Contam Part A Chem Anal Control Expo Risk Assess. 2019 Jan;36(1):141-151. doi: 10.1080/19440049.2018.1562237. Epub 2019 Jan 11. PMID: 30632916.

14. Tudi M, Atabila A, Ruan HD, Wang L, Lyu J, Tong S, Yu QJ. Natural dynamics and residues of pymetrozine for typical rice-growing areas of China. Ecotoxicology and Environmental Safety. 2022; 113230.

15. Chen Y, Ye H, Fang N, Luo Y, Wang X, Li Y, He H, Cheng Y, Zhang C. Residue Analysis and Dietary Risk Assessment of Pymetrozine in Potato (Solanum tuberosum L.) and Chrysanthemum morifolium (Ramat). Plants. 2023; 12(22): 3905. https://doi.org/10.3390/plants12223905

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