Food Anal. Methods (2016) 9:2471–2478
DOI 10.1007/s12161-016-0434-5

Fluorescence Polarization Immunoassay for Highly Efficient Detection of Imidaclothiz in Agricultural Samples

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Received: 5 November 2015 / Accepted: 1 February 2016 / Published online: 11 February 2016

Abstract A homogeneous fluorescence polarization immunoassay (FPIA) based on a monoclonal antibody for the detection of imidaclothiz was developed. Two fluorescein-labeled imidaclothiz tracers containing two different bridge lengths were synthesized and purified. Under optimal conditions, the 4-aminofluorescein-labeled imidaclothiz conjugate (AMF-labeled imidaclothiz), which contains a shorter bridge length, showed a higher sensitivity in the FPIA for detecting imidaclothiz, and the full analysis was achieved in less than 11 min. The IC_{50} and limit of detection (LOD, IC_{10}) were 87.94 ± 10.18 and 0.57 ± 0.16 μg/L, respectively. The spiked recoveries were 83 to 117 % measured in tomato, pear, rice, apple, cucumber, cabbage, and paddy water, with RSDs of 5 to 12 %. Furthermore, the results of FPIA for the authentic samples correlated well with those acquired by HPLC. Overall, the developed FPIA provided a simple, rapid, sensitive, and accurate method that was used for the quantitative detection of imidaclothiz in agricultural samples.

Keywords Imidaclothiz · Fluorescence polarization immunoassay · Monoclonal antibodies · Fluorescein-labeled imidaclothiz tracers

Introduction

Imidaclothiz, a member of the neonicotinoid insecticides with greater systemic activity, has been widely used for controlling whiteflies, thrips, leafhoppers, plant hoppers, and other various harmful pest species in China (Zhang 2005; Lan 2006; Xu et al. 2007). The action site locates insect nicotinergic acetylcholine receptors (Feng et al. 2008). It was developed and first commercialized by Nantong Jiangshan Agrochemical and Chemicals Co., Ltd. (China) (Dai 2005). In recent years, with the wide use of new neonicotinoid pesticides, the negative effects have become increasingly prominent. Neonicotinoid pesticides could affect honey bees' ability to collect nectar and could indirectly harm the pollinators (Henry et al. 2012). Therefore, monitoring the residual imidaclothiz in environmental and agricultural samples is significant.

The use of immunoassays as a rapid detection technology has been widely used for the detection of small molecules with many advantages, such as simplicity, low consumption, and high sensitivity. Recently, the detection of imidaclothiz using an indirect competitive enzyme-linked immunosorbent assay (ic-ELISA) has been reported (Fang et al. 2011). The simultaneous detection of imidaclothiz and other pesticides has also been developed later (Liu et al. 2013; Yan et al. 2014). However, ELISAs require multistep incubation and washing procedures, and such time-consuming technologies are unfit for the rapid detection of large samples. Therefore, it is necessary to develop a more rapid and convenient method to detect imidaclothiz.

Electronic supplementary material The online version of this article (doi:10.1007/s12161-016-0434-5) contains supplementary material, which is available to authorized users.

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The fluorescence polarization immunoassay (FPIA) is a potential homogeneous assay that can be considered as an alternative or complementary method for residue analysis with significant advantages, such as simplicity, rapidity, and short incubation times (Smith and Eremin 2008). FPIA is based on the competition between an analyte and a fluorescein-labeled tracer for binding antibody. The fluorescence polarization (FP) values will gradually decrease with increasing concentration of analyte. When there is no analyte, the fluorescein-labeled tracer will adequately bind with the special antibody, making the fluorescent polarization (FP) reach the maximum value. If the sample contains the analyte, the analyte will displace the tracer to bind with the antibody, and the FP values will decrease (Zhang et al. 2013; Shim et al. 2004; Mi et al. 2013). In recent years, FPIA methods have been developed for the analysis of small molecular compounds such as fumonisins, deoxynivalenol, and aflatoxins in grains (Maragos et al. 2001, 2002; Nasir and Jolley 2002), therapeutic drug levels in human plasma and mycotoxin ochratoxin A in food samples (Shim et al. 2004; Jolley 1981). An FP-based aptamer biosensor for detecting protein was also reported (Yue et al. 2014). Currently, a portable device that can detect FP quickly, the Sentry portable FP instrument, has been presented (Chun et al. 2009). It can be used for the on-site detection of an analyte.

This paper describes the development of rapid FPIA based on a monoclonal antibody (MAb) for the determination of imidaclothiz in agricultural samples. To optimize the FPIA, two fluorescein-labeled imidaclothiz conjugates were synthesized and investigated. Moreover, the FPIA was confirmed by HPLC. In this paper, a sensitive FPIA-based MAb for the detection of imidaclothiz was developed for the first time.

Materials and Methods

Reagents and Equipment

Imidaclothiz (97 %) was obtained from Nantong Jiangshan Agrochemical and Chemicals Co., Ltd. (Jiangsu, China). Bovine serum albumin (BSA), ovalbumin (OVA), fluorescein isothiocyanate (FITC) isomer I, Freund's complete and incomplete adjuvants, N-hydroxysuccinimide (NHS), N,N-dicyclohexylcarbodiimide (DCC), polyoxyethylene sorbitan monolaurate (Tween-20), and 4-aminofluorescein (4-AMF) were purchased from Sigma-Aldrich Chemical Co., Ltd (St. Louis, MO, USA). N,N-Dimethylformamide (DMF) and ethylenediamine dihydrochloride (EDA) were supplied by Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). The imidaclothiz hapten immunogen (hapten conjugated with bovine serum albumin), coating antigen (hapten conjugated with ovalbumin), and anti-imidaclothiz MAb were prepared and stored in the laboratory (Fang et al. 2011). All other chemicals used were of analytical reagent grade. Black microplates (96-well) were obtained from Corning Costar Corporation (NY, USA). The result of FPIA was detected using a Molecular Devices SpectraMax M5 (Sunnyvale, CA, USA). Imidaclothiz was detected using an Agilent 1260 HPLC Chromatograph (Agilent, USA).

Buffer and Solutions

Sodium borate buffer (BB, 0.02 mol/L, pH 7.4) was used for all FPIA experiments. Standard solutions of imidaclothiz and cross-reactants were prepared by the dilution of these compounds to 10 g/L in methanol and stored at 4 °C.

Fluorescein-Labeled Imidaclothiz Conjugate Preparation

To investigate the influence of the structure of fluorescein-labeled imidaclothiz conjugate, two tracers were synthesized with different bridge lengths (Fig. 1) and purified by TLC.

Fluorescein-labeled imidaclothiz conjugates with different bridge lengths were synthesized according to the method of Kolosova et al. (2003) with modifications. FITC was dissolved in a mixture of 1 mL of methanol and 0.01 mL of triethylamine; the solution was then added dropwise while stirring the solution of EDA (150 μmol of EDA in a mixture of 5 mL of methanol and 0.05 mL of triethylamine) in 30 min. The mixture was stirred for an additional 1 h at room temperature (RT) and kept overnight in the dark. The red-colored precipitates of fluorescein thiocarbamyl ethylenediamine (EDF) was filtered and dried at RT in the dark.

At the same time, 13.5 mg (40 μmol) of hapten was dissolved in 0.5 mL of DMF, and then 9.2 mg (80 mmol) of NHS and 16.5 mg (80 mmol) of DCC were added to the solution and stirred overnight at RT. After the reaction mixture was centrifuged to remove the precipitate, the resultant solution of hapten-NHS was added to 10 mmol of EDF (4-aminofluorescein can directly react with hapten-NHS), and the mixture was stirred at RT for 4 h in the dark.

The yellow reaction mixture solutions were separated by the TLC method using chloroform/methanol (4:1, *v*/*v*) as the eluent (Xu et al. 2011). The major yellow bands of varying R_f were collected, eluted with methanol, and stored at −20 °C in the dark. The specific binding of the synthesized tracers to the anti-imidaclothiz antibody was determined by evaluating the FP value of the mixture of tracers and antibody, and the structures of two tracers were identified by mass spectrometry.

FPIA

The optimum tracer concentration was achieved depending on the dilution, yielding a total fluorescence intensity signal that reached approximately 1000 fluorescence units

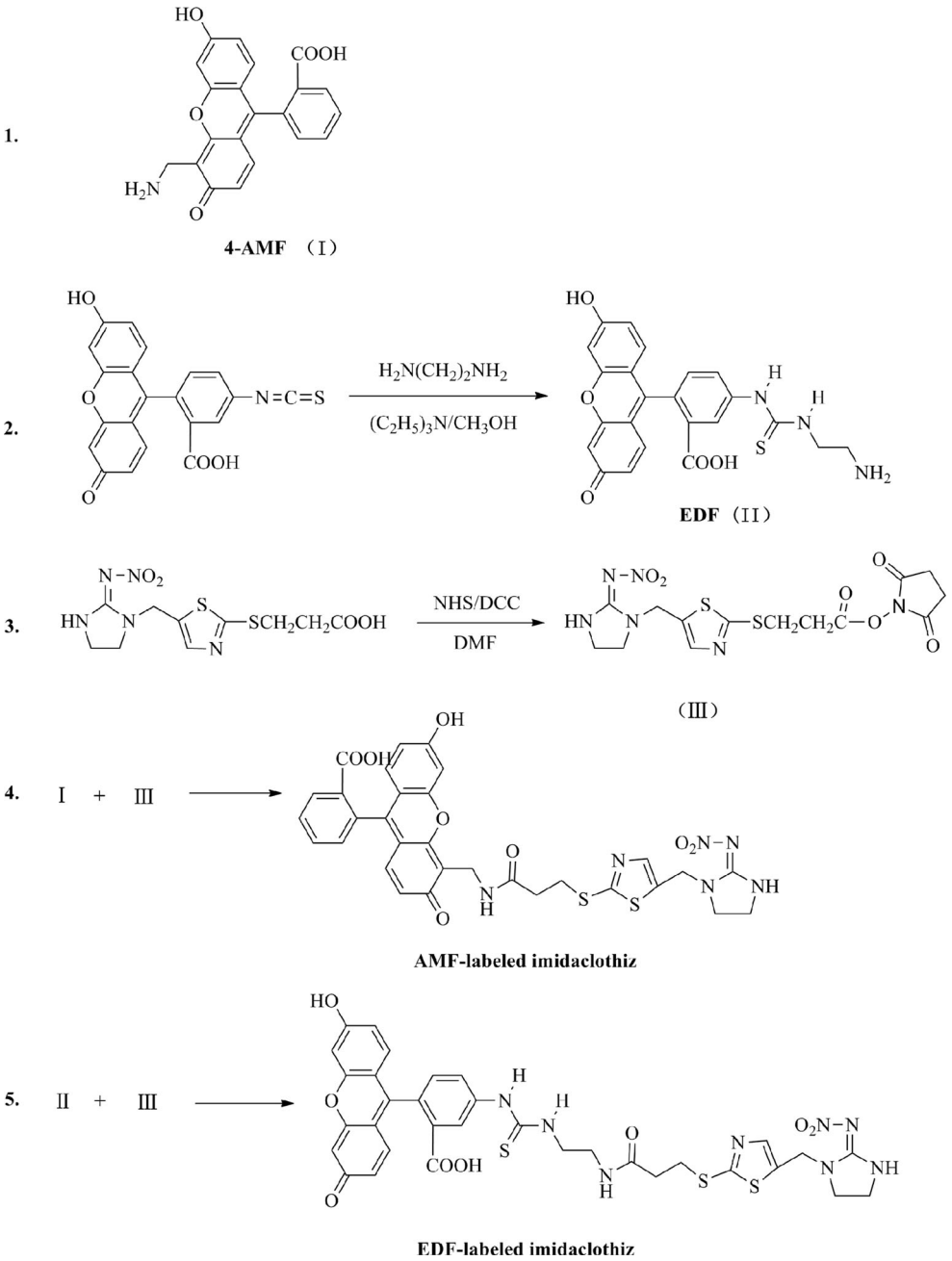


Fig. 1 Synthetic route for tracers and its structure. *III*: the synthesis of active ester based on imidaclothiz hapten

and an appropriate value of FP (Tang et al. 2008). For the optimal antibody concentration, a series of diluted antibody solutions were added to a certain amount of tracer, and FP values were recorded after a few minutes of incubation. The diluted solution, which could reach approximately 50 % of FP_{max}, was defined as the optimum concentration. Kinetics curves of FPIA were generated by recording the FP values ranging from after mixing the antibody with tracer to 15 min.

The inhibition curves based on the two tracers were established under the optimal tracer and antibody concentrations. The tracer having a lower IC_{50} value was selected for further optimization. The effect of methanol, an organic solvent commonly used in FPIA procedures to improve analyte solubility, was studied using BB containing 0, 5, 10, 20, 30, and 40 % methanol to dilute the imidaclothiz standard. BB buffers of different ionic strengths (0.1–0.6 mol/L Na^+) were tested to evaluate the effect of ionic strength. The effect of pH values was evaluated using different BB solutions ranging from pH 4.4 to 9.4. The evaluations were based on the maximum FP value (FP_{max}), the half-maximal inhibition concentration (IC_{50}), and the ratio of FP_{max}/IC_{50}. The combination of lower IC_{50} and higher FP_{max}/IC_{50} was the most desirable.

The FPIA was carried out using 96-well black microplates. Seventy microliters of tracer solution, 30 μL of imidaclothiz standard or sample solution of imidaclothiz, and 100 μL of antibody solution were added sequentially to the microplate for a total of 200 μL per well. The reaction mixture was incubated for 11 min at RT in the dark, and the values of FP were then measured. A standard curve for imidaclothiz was obtained under optimum conditions by plotting the percent binding of FP (% mP) versus the concentration of imidaclothiz using Origin Pro 8.0 software (OriginLab, MA, USA).

Cross-Reactivities

Cross-reactivities (CRs) for compounds that have a similar structure to imidaclothiz were determined using the optimized FPIA. The values of CRs were calculated according to the following formula:

$$CR\% = (IC_{50}\,\text{of imidaclothiz}/IC_{50}\,\text{of analogue}) \times 100$$

Analysis of Spiked Samples

The accuracy and precision of FPIA were evaluated by testing spiked samples. Tomato, pear, rice, apple, cucumber, cabbage, and paddy water that had been certified as free of imidaclothiz by HPLC (the limit of quantitation is 50 μg/L) were used for the recovery studies.

The filtered paddy water samples (10 mL) were spiked with imidaclothiz at 0.05, 0.5, and 2 mg/L and stored overnight; the samples were then directly analyzed by FPIA. The samples of tomato, pear, rice, apple, cucumber, and cabbage were homogenized fully. These samples (10 g) were added to imidaclothiz at concentrations of 0.05, 0.5, and 2 mg/kg and stored overnight. The samples were extracted twice by sonication in 10 mL of BB containing 50 % methanol for 10 min and then centrifuged at 4000 rpm for 10 min. The supernatant was adjusted to 25 mL with BB. After appropriate dilution, the solutions were analyzed via the FPIA.

Evaluation of Authentic Samples by FPIA and HPLC

Imidaclothiz WP (10 %) diluted with water was sprayed into authentic samples (including paddy water and pear). The paddy water samples were picked on 0, 1, 5, and 10 days after spraying, and pear samples were picked on 0, 5, and 10 days after spraying. The blank samples before spraying had been certified as free of imidaclothiz and imidacloprid by HPLC. The picked samples were analyzed by FPIA and HPLC. The extraction and dilution of FPIA followed the procedures of the spiked samples.

For HPLC, the filtered paddy water samples were extracted two times by 30 and 20 mL of acetonitrile. The acetonitrile was anhydrated and evaporated to dryness, and then 2 mL methanol and water (30:70, *v*/*v*) was added to the residue. The concentrated extract was confirmed by HPLC (Agilent 1260) with a DBX-C18 column (250 mm × 4.6 mm × 5 mm). The pear samples were mixed with 50 mL of acetonitrile by vortexing, extracted by sonicating for 10 min, and centrifuged at 4000 rpm for 10 min. The supernatant was then filtered through anhydrous sodium sulfate and concentrated. The concentrated extracts were subsequently diluted with 2 mL of methanol and water (30:70, *v*/*v*) and confirmed by HPLC. A mixture of methanol and water (30:70, *v*/*v*) was used as the mobile phase at a flow rate of 1.0 mL/min at 30 °C. The detection wavelength was 270 nm, and the injection volume was 20 μL.

Results and Discussion

Synthesis of the Fluorescein-Labeled Imidaclothiz Conjugates

The two reaction mixtures were purified by TLC, and three yellow bands were obtained from each reaction mixture (Fig. S1, see Supplementary Information (SI)). The results of FP verification demonstrated that the bands at $R_f = 0.3$ and $R_f = 0.5$ could be the tracers of AMF-labeled imidaclothiz and EDF-labeled imidaclothiz, respectively (Fig. S2, see SI). The verification result of ESI-MS for the two bands were as follows: AMF-labeled imidaclothiz, ESI-MS (positive) *m*/*z* 675.1 $[IMI\text{-}AMF + H]^+$, and EDF-labeled imidaclothiz, ESI-MS (positive) *m*/*z* 763.1 $[IMI\text{-}EDF + H]^+$. These results indicated that the tracers were synthesized successfully.

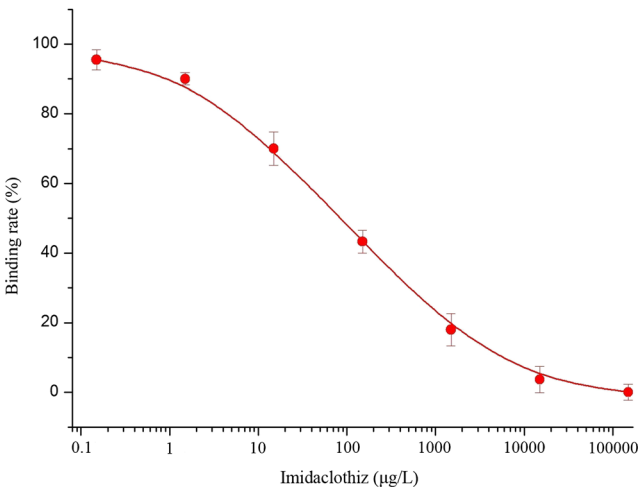
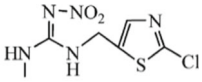
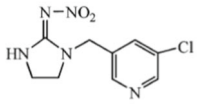
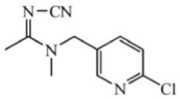
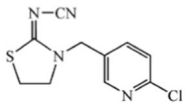
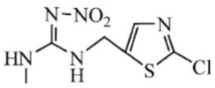
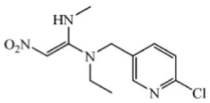
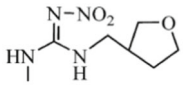
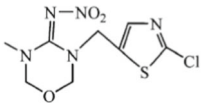


Fig. 2 FPIA calibration curves for imidaclothiz using AMF-labeled imidaclothiz with pH 7.4 BB buffer containing 5 % methanol and 0.4 mol/L Na^+ ($n = 3$)

Table 1 Cross-reactivity of imidaclothiz toward its analogues in FPIA

Compound	Structure	IC_{50} (μg/L)	CR (%)
Imidaclothiz		87.94	100
Imidacloprid		90.78	96.87
Acetamiprid		4128.64	2.13
Thiacloprid		8375.24	1.05
Clothianidin		19117.39	0.46
Nitenpyram		>100000	<0.1
Dinotefuran		>100000	<0.1
Thiamethoxam		>100000	<0.1

Optimization of FPIA

The assay parameters are important to enhance assay sensitivity. Improved sensitivity was obtained by choosing the antibody and tracer concentrations and the incubation time. The optimal concentrations of tracers were diluted by 400- and 200-fold for AMF- and EDF-labeled imidaclothiz, respectively. The antibody dilution curves for the two tracers are described in Fig. S3 (see SI). AMF-labeled imidaclothiz showed higher binding with the antibody, and the optimum antibody concentrations were 82.5 and 55 mg/L for AMF- and EDF-labeled imidaclothiz, respectively. Particular attention should be paid to the choice of incubation time (Chun et al. 2009; Li et al. 2014), as the kinetics curves (Fig. S4, see SI) revealed that the equilibrium was achieved 11 min after the mixing of antibody and tracer, demonstrating that the antibody and tracer had sufficiently bound.

The performances of tracers were further assessed by plotting an inhibition curve to obtain the IC_{50}. As shown in Fig. S5 (see SI), the IC_{50} of AMF-labeled imidaclothiz was 440.0 μg/L, which is higher than that of EDF-labeled imidaclothiz with an IC_{50} of 670.0 μg/L. It has been proven that AMF-labeled imidaclothiz exhibits a higher affinity to antibody and a higher sensitivity (lower IC_{50} values) than the tracer of EDF. Therefore, AMF-labeled imidaclothiz was a better tracer to perform FPIA. For FPIA, the tracers with different bridge lengths would result in different binding strength between tracer and the antibody, and then influence the sensitivity of FPIA (Chun et al. 2009).

Organic solvent, ionic strength, and pH were investigated to optimize the immunoassays. As demonstrated in Fig. S6

(see SI), the FP values decrease with increasing methanol content from 5 to 40 %. When the methanol content was 5 %, FPIA showed the highest FP_{max}/IC_{50}. The change in the Na^+ concentration from 0.1 to 0.6 mol/L clearly influenced the immunoassays. The change curve suggested that the FPIA showed the highest FP_{max}/IC_{50} at 0.4 mol/L. The IC_{50} values of FPIA were changed with pH values from 4.4 to 9.4, and the FPIA was more sensitive at pH 7.4. Therefore, the BB solution containing 5 % methanol and 0.4 mol/L Na^+ at pH 7.4 was chosen to perform FPIA to analyze imidaclothiz in order to obtain the best performance.

Sensitivity

The standard curve for imidaclothiz analyzed by FPIA using AMF-labeled imidaclothiz was obtained under optimal conditions (5 % methanol content, 0.4 mol/L Na^+, pH 7.4) (Fig. 2). The IC_{50}, LOD (IC_{10}), and linear range were 87.94 ± 10.18, 0.57 ± 0.16, and 0.57 μg/L to 90.9 mg/L, respectively. The LOD of the developed FPIA was approximately 1.2- to 10-fold lower than that of the previously reported FPIAs for melamine (Wang et al. 2011), zearalenone (Chun et al. 2009), and ochratoxin A (Shim et al. 2004). Based on the same MAb, the FPIA showed nearly the same sensitivity compared with indirect competitive ELISA (ic-ELISA) with an IC_{50} of 87.5 μg/L (Fang et al. 2011). However, ELISA is a heterogeneous method and requires a long period and multiple steps for analysis, making it difficult to meet the fast, simple demands on analytical screening (Shim et al. 2004; Mi et al. 2013). HPLC has been used successfully for the detection of imidclothiz with LOD of 0.4 μg/L (He et al. 2009). But, the method is time-consuming, laborious, and unsuitable for screening a large number of samples. Moreover, it often needs expensive instrumentation, large amounts of reagents, and complicated sample pretreatment (Li et al. 2014; Chun et al. 2009; Shim et al. 2004).

There is no suggested MRL of imidaclothiz in European Community (EC), China, and other standard regulations. But, the MRLs of clothianidin and thiacloprid which belong to neonicotinoid class have been established. In the USA, the MRLs of clothianidin are 10, 20, and 50 μg/kg for sorghum, beet, and potato, respectively (Li et al. 2012). The MRLs of thiacloprid have been assigned 20 μg/kg on cabbage in EC (http://ec.europa.eu/sanco_pesticides/public/index.cfm). Compared to the MRLs of neonicotinoid class, the sensitivity of the FPIA could meet the requirement for detecting imidaclothiz.

Specificity of FPIA

The cross-reactivity of the analogues of imidaclothiz was determined by performing a competitive assay and comparing the IC_{50} values. As shown in Table 1, the FPIA had a CR of 96.87 % for imidacloprid, and no CR was observed with the other analogues. The results were consistent with those of the ic-ELISA reported earlier (Fang et al. 2011). For this reason, imidaclothiz and imidacloprid have a similar imidazole ring and =N–NO_2, which likely played a significant role in the immunoreactions.

Table 2 Recovery of imidaclothiz spiked by FPIA

Sample	Spiked concentration (mg/L or mg/kg)	Mean recovery ± SD (%)	RSD (%)
Tomato	0.05	87 ± 8	9
	0.5	93 ± 8	8
	2	89 ± 9	9
Pear	0.05	96 ± 6	6
	0.5	108 ± 8	8
	2	90 ± 7	7
Rice	0.05	111 ± 9	9
	0.5	110 ± 6	5
	2	90 ± 8	8
Apple	0.05	106 ± 7	7
	0.5	105 ± 6	6
	2	113 ± 9	9
Cabbage	0.05	117 ± 12	11
	0.5	85 ± 8	9
	2	113 ± 9	9
Cucumber	0.05	86 ± 12	12
	0.5	116 ± 9	9
	2	87 ± 12	11
Paddy water	0.05	89 ± 5	6
	0.5	87 ± 12	12
	2	83 ± 11	12

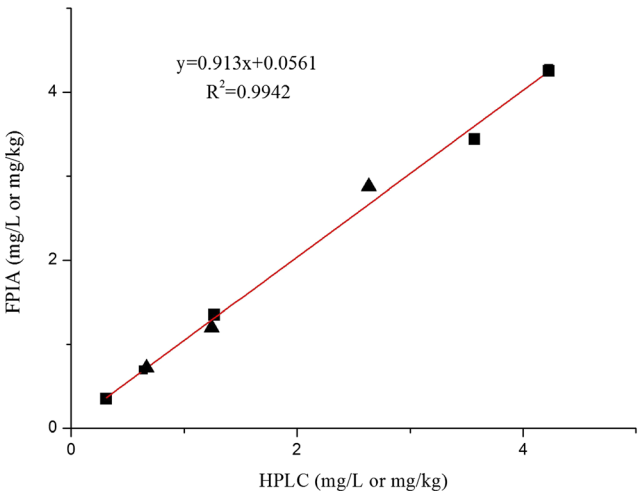


Fig. 3 Correlation between FPIA and HPLC for the blind samples. The *triangle* represents the concentration of imidaclothiz in pear samples; the *square* represents the concentration of imidaclothiz in paddy water samples

Recovery of Spiked Samples

Measurements can be affected by background fluorescence from the sample matrix in a homogeneous assay system. Dilution with buffers was the easiest and most immediate way to minimize or avoid matrix effects. The matrix effects of samples were studied by testing 5-, 10-, and 20-fold dilutions of sample extraction with BB. Figure S7 (see SI) demonstrates a 5-fold dilution of tomato, pear, rice, cucumber, and paddy water; a 20-fold dilution of apple and cabbage could remove the matrix effects. The recoveries ranged from 83 to 117 %, and the RSDs ranged from 5 to 12 %, indicating high accuracy and precision of the FPIA when applied to agricultural and environmental samples (Table 2).

Correlation of FPIA with HPLC

The representative chromatograms of HPLC indicated that the matrix interferences of paddy water and pear were removed by the sample treatments (Fig. S8, see SI). The accuracy and precision of the HPLC method was evaluated by measuring the spiked water and pear samples (the final concentrations were 0.1, 0.5, and 5 mg/L or mg/kg). The average recoveries ranged from 82 to 113 %, and the RSDs were less than or equal to 6 % (Table S1, see SI). The authentic samples of paddy water and pear contaminated with imidaclothiz were tested by both the developed FPIA and HPLC. As shown in Table S2 (see SI), the concentration of imidaclothiz in real samples as determined using the FPIA and HPLC were in the ranges 0.31–4.23 mg/L (mg/kg) and 0.35–4.27 mg/L (mg/kg), respectively. The results of FPIA and HPLC showed good correlations ($y = 0.9913x + 0.0561$, $R^2 = 0.9942$) (Fig. 3). The results indicated that FPIA is accurate and can be used to detect imidaclothiz in the authentic samples.

Conclusion

A rapid FPIA analytical method based on a MAb was developed for the detection of imidaclothiz. Two fluorescein-based imidaclothiz conjugates with different bridge lengths were synthesized and used as tracers to optimize the FPIA. The results demonstrated that the AMF-labeled imidaclothiz performed with higher sensitivity in the FPIA for detecting imidaclothiz. Imidaclothiz could be determined by the developed FPIA with a LOD of 0.57 ± 0.16 μg/L and an IC_{50} of 87.94 ± 10.18 μg/L. The assay was achieved in one step in 11 min. The spiked tests showed that the accuracy and precision of the FPIA met the requirements of residue analysis for imidaclothiz. Compared with other immunoassay methods, such as ELISA and time-resolved fluoroisnmunoassay (TRFIA), the developed FPIA offers a great improvement in shortening the overall testing time and analytical procedure. Moreover, it does not require expensive instrumentation in comparison to HPLC. In conclusion, the advantages of proposed FPIA make it a rapid, simple, and sensitive method for the quantitative detection of imidaclothiz in agricultural samples.

Acknowledgments This work was supported by the National Natural Science Foundation of China (31301690), the Doctoral Program of Higher Education Research Fund (20130097120006), and the Youth Innovation Fund in Science and Technology of Nanjing Agricultural University (KJ2013008).

Compliance with Ethical Standards

Conflict of Interest Ming Ma declares that he has no conflict of interest. Mo Chen declares that she has no conflict of interest. Lu Feng declares that she has no conflict of interest. Hongjie You declares that he has no conflict of interest. Rui Yang declares that she has no conflict of interest. Anna Boroduleva declares that she has no conflict of interest. Xiude Hua declares that he has no conflict of interest. Sergei A. Eremin declares that he has no conflict of interest. Minghua Wang declares that he has no conflict of interest.

Ethical Approval This article does not contain any studies with human participants or animals performed by any of the authors.

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