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ORIGINAL ARTICLE

Detection of the herbicide fenoxaprop-P-ethyl, its agronomic safener isoxadifen ethyl and their metabolites residue in rice

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Abstract

Introduction The herbicide fenoxaprop-P-ethyl and its agronomic safener isoxadifen ethyl, are used on cereals, and their as well as main metabolites residues can occur in rice. *Objectives* The present work aims to develop and to validate an analytical method for the determination of the herbicide, its safener and their metabolites in rice edible fractions. *Methods* Samples were extracted in acetonitrile (under acid conditions for active substances analysis); then, parent compounds were determined by gas chromatography with a mass spectrometer detector, while metabolites were analysed by liquid chromatography tandem mass spectrometry. *Results* The method was validated in rice straw, grain and plant: accuracy (mean recovery) was in the range 76–86% and 90–103% for parent compounds and metabolites, respectively, while precision (relative standard deviation) was in the range 3–11% and 6–17%. The limit of detection was 0.01 mg kg^{-1} for each analyte while limit of quantification was set to 0.05 mg kg^{-1}. Analysis of field collected samples enabled to remark differences. *Conclusion* The analytical method is suitable for the quantitative determination of each analyte considered in rice commodities.

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Introduction

Fenoxaprop-P-ethyl (ethyl (*R*)-2-[4-[(6-chloro-2-benzoxazolyl)oxy]phenoxy]propanoate, FPE) is an aryloxyphenoxypropionate post-emergence herbicide with contact and systemic action, which can be used on several crops against annual and perennial grasses. Nisha and Chopra (2005) reported the use of FPE on wheat, and McMullan (1994) described its use on barley. FPE use against rice grasses is also well documented by many authors (Bhattacharya *et al.*, 2001, 2004; Saini & Angiras, 2002). FPE persistence in environmental compartments is short (Han *et al.*, 1998; Guo *et al.*, 2008), while data concerning its magnitude of residues in edible commodities after field application are still missing. As far as concerns FPE degradation pathway, it is reported that the parent herbicide generates many metabolites (Song *et al.*, 2005), which are relevant to estimate its ecotoxicity to Daphnia Magna (Lin *et al.*, 2007). The two FPE main metabolites should therefore be considered when determining its magnitude of residues in edible commodities as well.

Additionally, commercial FPE formulations have to include an agronomic safener to avoid phytotoxicity symptoms which are described in cereals (Geminiani *et al.*, 2008). Isoxadifen ethyl (ethyl 4,5-dihydro-5,5-diphenyl-1,2-oxazole-3-carboxylate, IE) is an herbicide safener reported for maize (Rapparini & Fabbi, 2005) and for rice (Buehring *et al.*, 2001, 2006; Scherder *et al.*, 2001).

A reliable analytical method, suitable for the quantitative determination of the herbicide, its safener and their metabolites is not reported in literature yet, even if it could represent an important tool to further assess the fate of the residues after spraying and to evaluate human exposure of these chemicals through diet. Song *et al.* (2005) reported the separation of FPE biodegradation products by HPTLC, but this method had not quantification purposes, while Balinova (1996) described the analysis of fenoxaprop-ethyl and fenoxaprop in drinking water using solid-phase extraction and ion-pair HPLC and Celi *et al.* (1993) determined the same analytes in soil by reverse phase HPLC with UV detection after clean up on florisil or alumina cartridges.

These methods reported were limited because could not determine the safener and significant metabolites other than fenoxaprop, and because were not enough sensitive and specific; moreover, they were not tested for residues in vegetables. Hence, the present work was aimed to develop and validate an analytical method for parent compounds FPE and IE, as well as for their metabolites determination in/on rice plant, rice straw and rice grain. The FPE metabolites considered were 2-(4-(6-chloro-2-benzoxazolyloxy)-phenoxy)-propionic acid (fenoxaprop, FPE-M1) and 6-chloro-2,3-dihydro-benzoxazol-2-one (FPE-M2); these metabolites are known to be relevant in crop (Fenoxaprop-P-ethyl). The IE metabolites considered were, however, 5,5-diphenyl-2-isoxazoline-3-carboxilic acid (IE-M1) and 5-(4-hydroxyphenyl)-5-phenyl-2-isoxazoline-3-carboxilic acid (IE-M2) (Figure 1).

Rice (*Oryza sativa* L.) was chosen because this crop was assumed to be actually representative for the use of the chemicals considered.

Materials and methods

Method of analysis for parent compounds

Apparatus: (a) GC Agilent 6890 series gas chromatograph, equipped with: Agilent 5973i mass spectrometer (MS) detector and autosampler, interfaced to MSD ChemStation data acquisition and processing software – (Agilent Technologies, Santa Clara, CA, USA). (b) Fused silica capillary column (30 m × 0.25 mm i.d.), HP-5MS, film thickness 0.25 μm, GC column from Agilent (Agilent Technologies). (c) Chromatographic columns Chem-Elut CE 2050 (Varian Inc., Palo Alto, CA, USA).

Reagents: (a) Acetonitrile and hydrochloric acid, reagent grade from Merck (Darmstadt, Germany). (b) Cyclohexane+ethyl acetate (85+15 by volume) from Sigma Aldrich Chemical Co. (St. Louis, MO). (c) FPE certified reference standard, 99.5% pure, supplied by AgrEvo GmbH (Frankfurt, Germany). (d) IE certified reference standard, 99.7% pure, supplied by AgrEvo GmbH. (e) Atrazine certified reference standard, 99.8% pure, supplied by Sigma Aldrich Chemical Co.

Standard solutions: Stock solution was prepared by dissolving FPE and IE (25 mg, corrected for purity) reference standard in a flask with ethyl acetate (50 mL). Working standard solutions were prepared by diluting the stock solution until reaching a concentration of 0.03, 0.10, 0.50 and 1.0 $\mu g\,L^{-1}$ with the diluted internal standard solution.

Stock solution was prepared by dissolving atrazine (10 mg, corrected for purity) of reference standard in a flask with acetone (50 mL). Working standard solutions of atrazine were prepared by diluting the stock solution until reaching a concentration of 12 $\mu g\,mL^{-1}$ with ethyl acetate.

An accurate weigh of each sub-sample (25.0 g) was extracted by 80 mL (40 mL for rice grain) of acetonitrile +0.1 M hydrochloric acid (80+20 by volume) mixture, for 10 min on a shaker at room temperature. The suspension was filtered on a paper filter by a Buchner funnel into a round bottom flask, and the filter was washed twice with acetone (5 mL each). The filtrate was then loaded onto the ChemElut CE2050 column and eluted with cyclohexane+ethyl acetate (150 mL) mixture. The eluate, collected in a vacuum flask, was concentrated to dryness by a rotary evaporator. Rice straw extracts were concentrated directly after filtration without any clean up step. All the dry residues was reconstituted in the internal standard solution (1 mL) and transferred to a vial for analysis.

The extract (1 μL) was injected into the gas chromatograph and analysed at the following conditions:

(a) oven temperature: 100 °C for 2 min, from 100 to 250 °C at 10 $°C\,min^{-1}$ rate, and 250 °C hold for 10 min;
(b) flow rate of carrier gas (helium) 1 $mL\,min^{-1}$;
(c) injection mode splitless (split open 50 sec);
(d) source temperature 230 °C;
(e) quadrupole temperature 150 °C;
(f) MS acquisition: single ion monitoring:
  FPE *m*/*z* 288 and 290;
  IE *m*/*z* 294.

Quantification was done by the internal standard approach, and calibration performed for each batch of analysis, in duplicate at four concentration levels (Figure 1).

Method of analysis for metabolites

Apparatus: (a) HPLC Sciex liquid chromatograph (Perkin Elmer, Wellesley, MA, USA), equipped with electrospray interface and API300 mass spectrometer (Applied Biosystems, Foster City, CA, USA). (b) ODS column (250 mm × 4.5 mm), Lichrocart Purospher RP18 (Merck). (c) Bakerbond SPE Florisil cartridges 500 mg (JT Baker, Phillipsburg, NJ, USA).

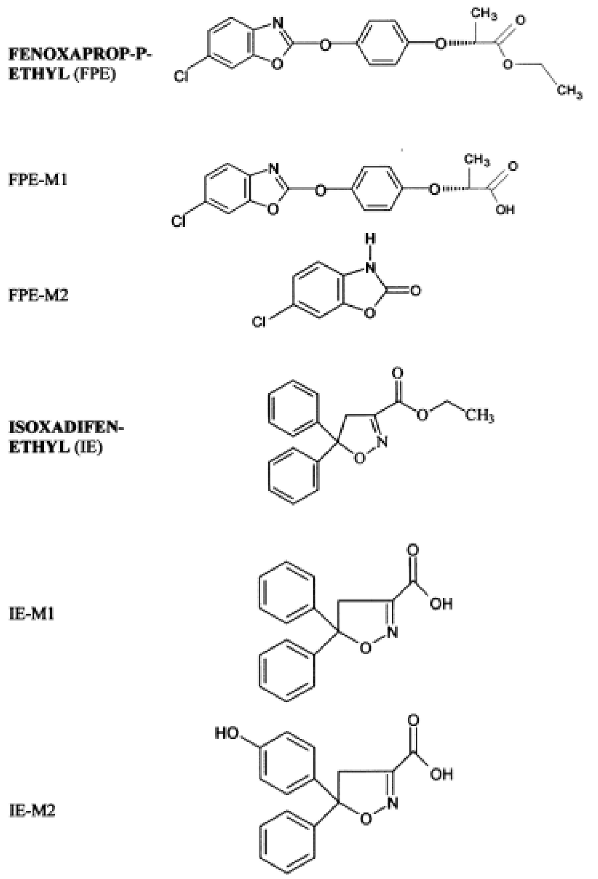

Figure 1 Parent compounds and metabolites structure.

Reagents: (a) Acetonitrile, HPLC grade from Merck. (b) Formic acid from Fluka (St. Gallen, Switzerland). (c) FPE metabolites FPE-M1 and FPE-M2 certified reference standard, 96.2% pure, supplied by AgrEvo GmbH. (d) IE metabolites IE-M1 and IE-M2 certified reference standard, 96.2% pure, supplied by AgrEvo GmbH.

Stock solution was prepared by dissolving each analyte (25 mg, corrected for purity) reference standard in a flask with acetonitrile (50 mL). Working standard solutions were prepared by diluting the stock solution until reaching a concentration of 0.03, 0.10, 0.50 and 1.0 μg mL^{-1} in acetonitrile.

An accurate weigh each comminute sub-sample (5.0 g) was extracted three times by 100+100+50 mL (50+50+30 mL for rice grain) of acetonitrile, for 3 min each on a shaker at room temperature. The suspension was filtered on a paper filter by a Buchner funnel into a round bottom flask, and the filter was washed with acetonitrile (8 mL). The filtrate was then concentrated to dryness by a rotary evaporator. The dry residue was reconstituted in acetonitrile (1 mL) and loaded onto a Florisil SPE cartridge washed previously with acetonitrile (10 mL). The cartridge was washed with acetonitrile (1 mL) and the metabolites eluted with further acetonitrile (3 mL). The purified extract was concentrated to dryness by a gentle stream of nitrogen and the dry residue reconstituted in acetonitrile (2 mL) to be analysed.

The extract (50 μL) was injected into the liquid chromatograph and analysed at the following conditions:

(a) mobile phase: acetonitrile (A) and 0.01 M formic acid aqueous solution (B), flow rate 0.9 mL min^{-1};
(b) mobile phase gradient: 85% A until 0.2 min, 90% A at 3 min, 85% A at 15 min;
(c) MS conditions: electrospray, polarity positive, nebulizer 14, current 8;
(d) MS acquisition: multiple reaction monitoring (Table 1).

Quantification was done by the external standard approach, and calibration performed for each batch of analysis, in duplicate at four concentration levels.

Methods validation

Recovery tests were conducted by analysing untreated samples from each matrix (rice plant, rice straw and rice grain) spiked with standard solutions, according to both the methods described previously. Concerning the determination of the parent compounds, two spiking levels were chosen and five replicate recoveries were determined per level and per each matrix. Validation tests for the metabolites were instead comprised of five replicate recoveries per level and per analyte; three of these five replicate were carried out on rice grain, while the remaining two were done on rice straw. Accuracy was expressed as mean recovery for each matrix, with five replicates both at 0.05 and 0.50 mg kg^{-1}.

Precision was evaluated for each matrix, at each spiking level, and expressed as Relative Standard Deviation (RSD), with five replicate analyses in sequence by the same operator in two different days (repeatability conditions).

The limit of detection, however, was calculated on the signal-to-noise basis: the baseline in the unfortified blank

Table 1 Tandem MS determination of FPE and IE metabolites

Analyte	Molecular weight	Parent ion (*m/z*)	Product ion (*m/z*)	CAD gas
FPE-M1	333.7	334.5	288.3	3
FPE-M2	169.6	171.2	46.1	3
IE-M1	267.1	267.9	45.9	3
IE-M2	283.1	283.9	220.2	3

FPE, fenoxaprop-P-ethyl; IE, isoxadifen ethyl; MS, mass spectrometer.

extract at the analyte retention time window was magnified to obtain the tallest (height) or broad (area) noise, which was multiplied by a factor three and then converted into concentration. The limit of quantification was instead assumed as the lowest spiking level at which both accuracy and precision were acceptable.

Analysis of field samples

The analytical method was finally applied to rice samples gained from a field trial performed in Pavia, Northern Italy. The formulation sprayed was an emulsion concentrate containing 69 g L^{-1} of FPE and 75 g L^{-1} of IE, and was applied once at tillering (BBCH 22–24). The field application rate was 1.25 L ha^{-1} to a plot, and 1.5 L ha^{-1} to a second experimental plot. Rice plants were collected few hours after spraying, while straw and grain were harvested at normal commercial harvest, 107 days after application. All the sampling events were carried out randomly on each plot, and threshing was done manually. At each sampling date, a control sample was also taken from an untreated area, to be used as reference matrix. Each sample was immediately comminute by a blender using dry ice, and then frozen, to be next analysed in triplicate according to the analytical methods described previously.

Results and discussion

The MS spectrum of both spiked and field-treated samples was consistent with the MS spectrum of standard solutions in solvent; additionally, unspiked rice plant, straw and grain sample extracts had no detectable residue and therefore they did not show any chromatographic interference. The method can therefore be considered specific enough.

As far as concerns the linear dynamic range, the correlation coefficient in all cases was well higher than 0.99, hence demonstrating an adequate correlation between instrumental response and concentration.

Regarding safety considerations, the reagents used were not highly hazardous, and therefore special precautionary handling procedures were unnecessary.

Accuracy and precision

The whole pool of recoveries gained from the analytical procedures described previously is presented in Tables 2 and 3, for parent compounds and metabolites, respectively. The values reported are the average of duplicate instrumental determinations. As far as concerns accuracy, the individual recoveries for FPE and IE were in the range 69.8–109.4%, while those for metabolites were found in the range 71.7–110.4%. The mean recoveries, for each analyte, were instead in the range 76–86% and 90–103% for parent compounds and metabolites, respectively. Precision was, however, in the range 3–11% and 6–17% for parent compounds and metabolites, respectively. The best precision (as lowest RSD) was achieved for FPE determination in rice straw, whereas the highest RSD was found for IE-M1 analysis.

Table 2 Parent compounds recovery tests

		Isoxadifen ethyl		Fenoxaprop-P-ethyl	
Matrix	Spiking level (mg kg^{-1})	Residue found (mg kg^{-1})	Recovery (%)	Residue found (mg kg^{-1})	Recovery (%)
Rice plant	0.05	0.040	79.3	0.041	81.1
	0.05	0.038	76.0	0.039	77.8
	0.05	0.053	106.0	0.036	72.9
	0.05	0.043	86.8	0.041	82.0
	0.05	0.040	80.2	0.042	83.1
	0.50	0.440	87.9	0.397	79.4
	0.50	0.474	94.7	0.349	69.8
	0.50	0.463	92.6	0.356	71.1
	0.50	0.381	76.1	0.363	72.5
	0.50	0.356	71.1	0.354	70.7
Mean			85		76
RSD			11		5
Rice straw	0.05	0.042	84.6	0.039	78.9
	0.05	0.038	75.5	0.040	80.1
	0.05	0.046	91.1	0.038	76.1
	0.05	0.035	70.8	0.037	73.7
	0.05	0.040	79.3	0.038	76.1
	0.50	0.357	71.4	0.371	74.1
	0.50	0.400	80.0	0.400	80.0
	0.50	0.365	72.9	0.386	77.1
	0.50	0.496	99.2	0.367	73.3
	0.50	0.373	74.5	0.371	74.2
Mean			80		76
RSD			9		3
Rice grain	0.05	0.044	88.1	0.049	98.8
	0.05	0.047	93.3	0.035	70.4
	0.05	0.041	82.9	0.046	92.0
	0.05	0.039	77.6	0.048	95.5
	0.05	0.055	109.4	0.048	95.9
	0.50	0.356	71.2	0.360	72.0
	0.50	0.373	74.5	0.353	70.6
	0.50	0.443	88.5	0.404	80.7
	0.50	0.438	87.6	0.404	80.7
	0.50	0.419	83.7	0.422	84.4
Mean			86		84
RSD			11		11

RSD, relative standard deviation.

Globally, both accuracy and precision were considered satisfactory, in compliance with European Commission method requirements for registration purposes [EU SANCO (Directorate General Health and Consumer Protection)].

Table 3 Parent compounds metabolites recovery tests

Analyte	Matrix	Spiking level (mg kg^{-1})	Residue found (mg kg^{-1})	Recovery (%)
FPE-M1	Rice grain	0.05	0.053	105.5
	Rice grain	0.05	0.052	104.4
	Rice grain	0.05	0.053	105.9
	Rice straw	0.05	0.050	100.7
	Rice straw	0.05	0.052	104.2
	Rice grain	0.50	0.535	107.0
	Rice grain	0.50	0.439	87.8
	Rice grain	0.50	0.500	100.0
	Rice straw	0.50	0.545	108.9
	Rice straw	0.50	0.525	104.9
Mean				103
RSD				6
FPE-M2	Rice grain	0.05	0.051	102.9
	Rice grain	0.05	0.052	103.5
	Rice grain	0.05	0.055	110.2
	Rice straw	0.05	0.044	87.4
	Rice straw	0.05	0.046	92.2
	Rice grain	0.50	0.434	86.7
	Rice grain	0.50	0.465	93.0
	Rice grain	0.50	0.458	91.6
	Rice straw	0.50	0.372	74.4
	Rice straw	0.50	0.524	104.8
Mean				95
RSD				11
IE-M1	Rice grain	0.05	0.055	110.1
	Rice grain	0.05	0.043	86.9
	Rice grain	0.05	0.054	108.2
	Rice straw	0.05	0.036	72.9
	Rice straw	0.05	0.040	80.4
	Rice grain	0.50	0.521	104.1
	Rice grain	0.50	0.397	79.3
	Rice grain	0.50	0.359	71.7
	Rice straw	0.50	0.531	106.2
	Rice straw	0.50	0.402	80.4
Mean				90
RSD				17
IE-M2	Rice grain	0.05	0.046	92.7
	Rice grain	0.05	0.054	108.5
	Rice grain	0.05	0.055	110.4
	Rice straw	0.05	0.045	89.0
	Rice straw	0.05	0.045	89.6
	Rice grain	0.50	0.451	90.2
	Rice grain	0.50	0.515	102.9
	Rice grain	0.50	0.517	103.4
	Rice straw	0.50	0.551	110.1
	Rice straw	0.50	0.542	108.4
Mean				101
RSD				9

FPE, fenoxaprop-P-ethyl; IE, isoxadifen ethyl; RSD, relative standard deviation.

Detection limits

The limit of detection was found to be 0.01 mg kg^{-1} for each analyte in all matrices; being accuracy and precision satisfactory at the lowest spiking level (0.05 mg kg^{-1}), this level was instead assumed as limit of quantification of the method. These limits, when compared with EU maximum residue limit in cereals grain (0.1 mg kg^{-1} as set out in the Regulation 149/2008 concerning harmonization of residue levels in EU countries) can be considered as adequate.

Statistics

Analysis of variance (ANOVA at 99% confidence level) evidenced that FPE recoveries were not different from IE recoveries and that method performance was not significantly different between rice straw, rice grain and rice plant matrices. Similarly, the performance of the method for metabolites determination was not significantly different concerning both matrices and target analytes.

The method fit with the purpose for which was developed, and could therefore be used either for monitoring or in residue trials for regulatory purposes.

Analysis of field samples

Any residue could be detected in the field samples gained from the untreated plot. Parent compounds residues in rice samples gained from the treated plot were, however, detected as a function of field dosage: FPE was 8.96 and 14.22 mg kg^{-1} while IE was 5.98 and 8.13 mg kg^{-1} at the lower and higher application rate, respectively. As far as concerns the FPE metabolites, FPE-M1 was the only compound detected at harvest, in the range 0.61–0.71 mg kg^{-1} in grain and from not detectable to 0.08 mg kg^{-1} in straw. IE metabolites were not detected in all the samples.

The results were in agreement with the information given by literature (Hoagland & Zablotowicz, 1998; Song *et al.*, 2005; Lucini & Molinari, 2010) concerning the degradation route and fate of FPE in environmental compartments: the herbicide residue decreased in time and was associated to the parallel increase of fenoxaprop-P (FPE-M1) residue. The metabolite fenoxaprop-P is the acid resulting from the hydrolysis of active ingredient propanoate ester bonding and this conversion was actually observed in the field trial performed on rice.

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