Quality control of seed treatments using near infrared spectroscopy

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Introduction

Seed treatment with plant protection products can achieve its objective only if the active substance is applied at the target rate and uniformly distributed between seeds of a same batch. The quality of seed treatments can accurately be determined by gas chromatography (GC) or high performance liquid chromatography (HPLC), but these methods are destructive, expensive, time consuming and therefore inapplicable for monitoring, especially for seeds harvested and treated a few time before sowing.[1–3]

This research investigates the possibilities of near infrared spectroscopy (NIR) to determine the quality of seed treatments, in order to allow a faster, less expensive and non destructive average quantification and also to evaluate the distribution of plant protection products between seeds of a same batch.

Materials and methods

NIR instrument

NIR was developed to analyse tefluthrin (a pyrethroid insecticide) on wheat seeds and imidacloprid (a neonicotinoid insecticide) on barley seeds. The NIRS analyses were carried out using a Foss NIRSystems 6500 spectrometer. The seeds spectra collected from 400 to 2500 nm were acquired in reflection mode. Two types of sample presentation were used: the "bulk presentation" (Figure 1) to determine the average active substance concentration in a seeds batch and the "single seed presentation" (Figure 2) to determine the active substance on individual seeds in order to evaluate the distribution of the seed treatment. In the "bulk presentation", the cell is rectangular and can contain 100 g of seeds. In the "single seed presentation", the seed is measured in a rotating drawer and the cell is equipped with a full aluminium disc with a cavity in its middle where the seed is placed. In both cases each sample was measured in duplicate. The spectral data were treated using the WINISI 1.5 software (Infrasoft International LLc, USA). The calibration equations were obtained by a modified partial least squares (MPLS) regression technique.

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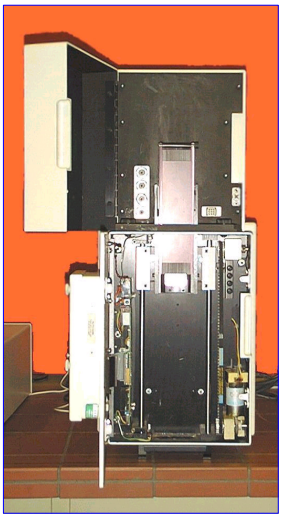

Figure 1. Foss NIRSystems 6500 : bulk presentation.

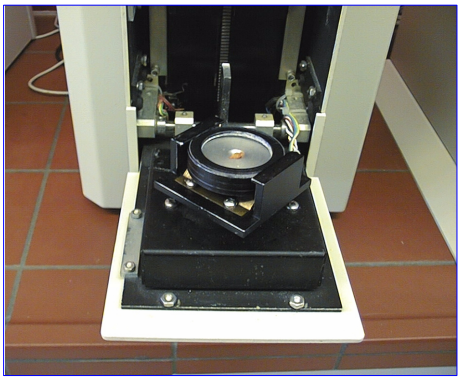

Figure 2. Foss NIRSystems 6500 : single seed presentation; rotating aluminium cup with one seed in the hole in the middle.

Seeds samples and chemical determinations

Samples of wheat seeds treated with tefluthrin and samples of barley seeds treated with imidacloprid were used to build the equations. These samples were coming from seeds industrial treaters or were treated in-house using a laboratory apparatus for seed treatment (HEGE 11). The samples of wheat seeds were analysed for tefluthrin content by gas chromatography with flame ionisation detection (GC-FID) or high performance liquid chromatography with diode array UV/visible spectrophotometry Detection (HPLC-DAD) after extraction with appropriate solvent. Samples of barley seeds were analysed for imidacloprid content by high performance liquid chromatography with diode array UV/visible spectrophotometry detection (HPLC-DAD) after extraction with appropriate solvent.

Some of these samples were also analysed for active substance concentration on individual seeds in order to evaluate the distribution of active substance among the seeds batch.

Results and discussion

Tefluthrin on wheat seeds

For the average tefluthrin concentration and distribution of treatment in a seeds batch, the best treatment of the spectral data is 2,5,5 (two for the second derivative, five for the gap and smoothing segment expressed in data points, respectively) without any scatter correction. Table 1 shows the performance of the calibration equation and of the prediction of totally independent samples (new year of production). The best equation presents a *SD*/*SEC* ratio of six. Figure 3, showing the scatter plot of the regression to predict the average tefluthrin concentration, indicates a good correlation between NIR values and reference values.

The Figure 4 shows the comparison between NIRS and reference method for the distribution of tefluthrin on 100 individual seeds of a same batch. Results obtained with the single seed

measurements are not so accurate than the bulk measurements but show nevertheless that NIRS allows a good estimate of the homogeneity of the seed treatment.

Table 1. Average tefluthrin concentration on wheat seeds: performance of the calibration equation and of the prediction of independent samples.

Calibration	*N*c	Mean	*SD*	Range	*SEC*	R^2c	*SECV*	R^2v	Terms	Scatter	Math
g/100 kg	90	16.7	7.4	4.7–34.2	1.3	0.97	1.8	0.94	7	None	2,5,5

Validation	*N*v	Mean	*SD*	Range	*SEP*	R^2p	bias	slope	*SEP*(*C*)
g/100 kg	18	9.5	3.1	6.2–15.4	2.5	0.89	–2.3	0.92	1.1

Nc = Number of samples for the calibration, SD = standard deviation,SEC = standard error of calibration
R²c = determination coefficient of calibration, SECV = standard error of cross validation, R²v = determination coefficient of cross validation
Nv = number of samples for the independent validation, SEP = standard error of prediction, R²p = determination coefficient of prediction

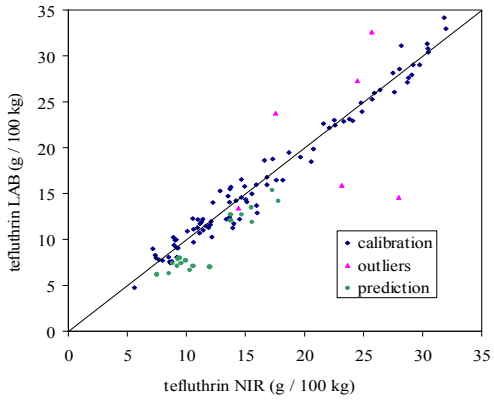

Figure 3. Average concentration of tefluthrin on wheat seeds.

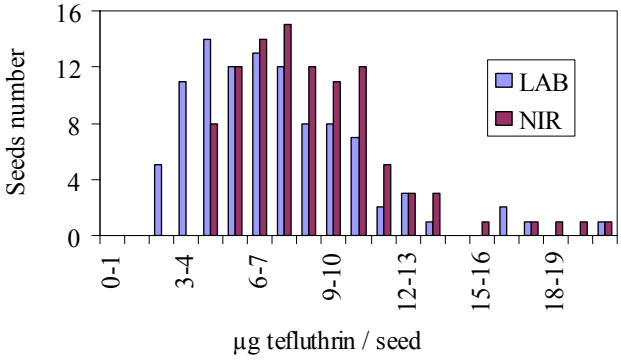

Figure 4. Distribution of tefluthrin on 100 individual wheat seeds of a same batch.

Imidacloprid on barley seeds

For the average imidacloprid concentration and distribution of treatment in a seeds batch, the best treatment of the spectral data is 2,5,5 (two for the second derivative, five for the gap and smoothing segment expressed in data points, respectively) with a detrend correction . Table 2

shows the performance of the calibration equation and of the prediction of totally independent samples (new year of production). The best equation presents a *SD*/*SEC* ratio of 8. Figure 5 showing the scatter plot of the regression to predict the average imidacloprid concentration indicates a good correlation between NIRS values and reference values.

The Figure 6 shows the comparison between NIRS and reference method for the distribution of imidacloprid on 100 individual seeds of a same batch. Results obtained with the single seed measurements are not so accurate than the bulk measurements but show nevertheless that NIRS allows a good estimate of the homogeneity of the seed treatment.

Table 2. Average imidacloprid concentration on barley seeds : performance of the calibration equation and of the prediction of independent samples.

Calibration	*N*c	Mean	*SD*	Range	*SEC*	R^2c	*SECV*	R^2v	Terms	Scatter	Math
g/100 kg	73	50.5	21.4	0.0–104.0	2.7	0.98	3.8	0.97	7	DET	2,5,5

Validation	Nv	Mean	*SD*	Range	*SEP*	R^2p	bias	slope	*SEP*(*C*)
g/100 kg	25	57.2	25.7	15.9–102.1	4.3	0.97	–0.1	1.04	4.4

Nc = Number of samples for the calibration, SD = standard deviation, SEC = standard error of calibration
R²c = determination coefficient of calibration, SECV = standard error of cross validation, R²v = determination coefficient of cross validation
Nv = number of samples for the independent validation, SEP = standard error of prediction, R²p = determination coefficient of prediction

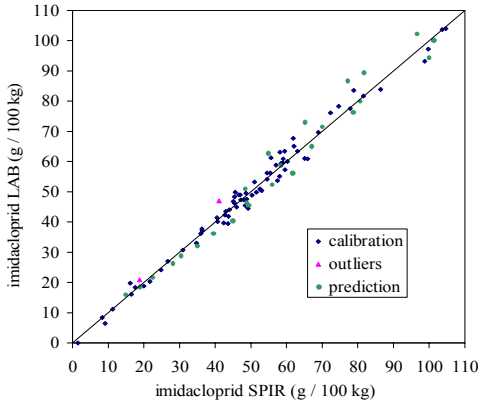

Figure 5. Average concentration of imidacloprid on barley seeds.

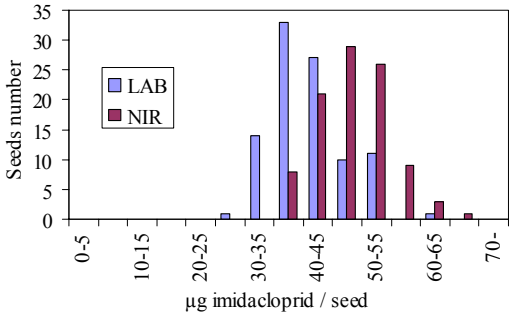

Figure 6. Distribution of imidacloprid on 100 individual barley seeds of a same batch.

Conclusions and further prospects

The performance characteristics of the calibration equations and the validation results obtained with tefluthrin on wheat seeds and imidacloprid on barley seeds prove that NIRS can determine quantitatively the average active substance concentration on a treated seeds sample with a good accuracy and also estimate the homogeneity of the seed treatment. This new application of NIRS offers new further prospects for the quality control of seed treatments with plant protection products.

NIRS has to be developed for other active substances of plant protection products and other seed species. Being a non destructive, rapid, accurate and low-priced method, NIRS is a powerful technique which can be used for the monitoring of seeds treated with plant protection products. This technique offers an interesting tool for agrochemical industry, seed industry, seed treaters, farmers, searchers involved in seed treatment and authorities.

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