USE OF NIR SPECTROSCOPY TO DETERMINE QUALITY FACTORS IN HARVEST SURVEYS OF CANOLA

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ABSTRACT

A whole-seed scanning NIR Spectrometer was used to determine oil, protein, glucosinolates, and chlorophyll in samples of canola seed from harvest surveys. Results of prediction sets and estimates of crop district averages, particularly for oil and protein content improved as samples from new year's surveys were added to the calibration. Glucosinolate prediction was hampered because of a lack of range in the prediction set. Selection of high and low samples from growing areas across Western Canada provided the best calibration/prediction set.

INTRODUCTION

The ability to quickly analyze oil, protein, chlorophyll and glucosinolates in a large number of samples of canola seed make NIR a valuable technique for determining the quality of canola seed in harvest surveys conducted by the Canadian Grain Commission. Until recently, it has only been able to analyze composite samples (by grade and crop district) of the 2000 to 4000 total samples generated in this survey. Analysis of individual samples would provide an indication of the range and distribution of individual quality factors across grade and crop district.

Earlier studies at the GRL had shown that whole seed NIR spectroscopy utilizing a NIRSystems 6500 analyzer was capable of determining oil content, protein content chlorophyll content and glucosinolates with a reasonable degree of accuracy to use the technique in surveys (Daun et al, 1994). This study shows how this technique has been applied to the Grain Research Laboratory's harvest survey of canola over the years 1992 to 1994.

EXPERIMENTAL

Samples of canola seed were received from country elevators and from Canadian Crushing plants as part of the Canadian Grain Commission's annual Harvest Survey for Canola (DeClercq *et al.*, 1992, 1993, 1994). Samples were cleaned and graded and the full spectrum scan (400 nm to 2400 nm) was obtained for all samples on a NIRSystems 6500 instrument operating in the reflectance mode.

Survey composites and prediction/calibration samples were analyzed according to the methods described by DeClercq *et al.* 1992. These methods include oil content by continuous wave NMR spectrometry (calibrated against F.O.S.F.A. oil extraction method),

protein as %N x 6.25 by combustion, chlorophyll by the ISO extraction spectrometric method and glucosinolates by HPLC (ISO Method). All results in this paper are presented on a moisture-free basis.

The NIR instrument was calibrated using PLS regression in the NSAS software package. In 1992, the instrument was calibrated using 100 samples selected from the first samples received in the harvest survey. The calibration was later adjusted using samples selected from throughout the survey and this calibration was used in 1993. A further adjustment to the calibration was made after the 1993 survey and this calibration was used in 1994 but on a different NIRSystems 6500 instrument than was used in the previous two years.

Assessment of Accuracy and Precision

Accuracy and precision were assessed both on prediction sets of samples drawn from the survey samples and from comparison of the crop district and grade means from NIR analysis with the analysis of composite samples. If the NIR method is to be used to replace the compositing system for determination of oil, protein etc., it is important that results from both methods be comparable.

In 1992 and 1993, prediction samples were drawn at regular intervals as samples arrived in the laboratory. This process gave a random sampling, but did not result in a uniform range of results. In 1994, prediction samples were selected from the high and low oil content samples from each provincial crop district. In addition, samples from each province and species were drawn to fill in any gaps between the high and low samples. The strong inverse correlation between oil and protein ensured that the set of samples covered the range for both parameters.

TABLE 1. Parameters for Prediction Samples

		Prediction Set				Crop District Composites			
	Year	Mean	Min.	Max.	Std. Dev.	Mean	Min.	Max.	Std. Dev.
Oil Content	1994	47.0	34.5	53.6	4.89	46.7	44.0	48.5	0.90
%, Dry Basis	1993	47.0	39.0	53.7	3.00	45.9	40.5	48.7	2.30
	1992	46.1	38.3	53.1	2.60	45.8	41.0	49.1	1.40
Protein Content	1994	22.3	16.5	31.9	3.91	22.1	19.9	25.0	1.00
%N x 6.25,	1993	21.9	17.3	29.9	3.00	21.7	19.1	26.2	1.70
Dry Basis	1992	22.8	15.8	30.6	2.80	22.8	18.7	25.5	1.40
Chlorophyll	1994	17.8	1.2	125.6	17.39	14.4	4.9	42.5	8.60
mg/kg	1993	24.2	1.2	82.0	19.30	23.7	11.1	50.0	10.40
	1992	16.1	0.0	55.4	12.60	21.6	6.7	40.0	9.40
Glucosinolates	1994	5.1	32.2	13.3	5.70	12.6	9.0	22.0	2.70
uM/g	1993	12.4	3.7	28.7	5.10	13.0	8.0	22.0	3.30
	1992	ND	ND	ND	ND	13.4	7.9	21.9	2.40

Glucosinolates were not determined on the prediction samples for 1992. In the other two years of the study, the standard deviation of glucosinolates for the prediction set (and for the crop district composites) was small, resulting in poor correlation.

Table 2. Statistics for Prediction Data.

Year	N	R2	Std. Dev.	RPD	N	R2	Std. Dev.	RPD
	Prediction Set				Crop District Composites			
Oil Content								
1994	74	0.961	0.58	8.51	32	0.948	0.21	4.45
1993	94	0.871	1.10	2.79	35	0.85	0.80	2.70
1992	230	0.893	0.80	3.84	56	0.81	0.60	2.06
Protein Content								
1994	74	0.985	0.49	8.12	32	0.916	0.30	3.50
1993	94	0.905	0.91	3.26	35	0.986	0.20	8.60
1992	230	0.945	0.60	4.91	56	0.849	0.50	2.40
Chlorophyll Content								
1994	74	0.726	6.21	1.49	32	0.919	2.43	3.58
1993	94	0.871	6.94	2.80	35	0.873	3.72	2.85
1992	230	0.691	2.70	3.10	56	0.803	7.30	2.10
Glucosinolates								
1994	74	0	8.00	0.00	32	0.347	2.12	1.28
1993	94	0.516	3.50	1.45	35	0.241	2.80	1.18
1992	230	ND	ND	ND	56	0.001	2.40	0.70

[a] Ratio of Standard Deviation of the Prediction Set to the Standard Error of Prediction.

The standard deviation for the crop survey composites where the NIR values was derived from a mean of a large number of values, was much smaller than the standard deviation for the prediction sets in all years and, at least in 1994, was close to the error expected from the wet chemistry. Standard errors and RPD values improved as further samples were added to the calibration set. It is notable that for 1994, there was need to apply a polynomial correction to the data for the prediction set for oil content. This is not unusual when data has been extended beyond the normal range of calibration.

The data suggest that NIR Spectroscopy will provide an accurate estimation of the oil, protein and chlorophyll content of crop districts provided that the number of samples averaged to provide each crop district mean is large.

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