ANALYSIS OF RAPESEED CHEMICAL COMPOSITION BY NIR TECHNIQUE

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INTRODUCTION

The standard methods for determination of chemical composition of oilseeds are so much time consuming processes for the monitoring of oilseed quality. Moreover, the sample is destroyed, which is an inconvenience for plant breeders, as some of the methods involve the use of flammable and hazardous chemicals. These serious drawbacks resulted in the development of both simple tests and instrumental techniques such as nuclear magnetic resonance (NMR) and near-infrared reflectance (NIR) spectroscopy for oilseed quality control.

The NIR technique has become established as a rapid and effective method for the wide range of analytical application. NIR has been used for determining not only basic chemical composition in cereal grains and oilseeds (Williams, 1975; Kaffka et al., 1982; Robertson and Barton, 1984/ but also e.g. amino acids (Williams et al., 1984) and gossypol (Brith and Ramey, 1982).

The present study was undertaken to examine the possibility of applying the NIR technique for the testing of rapeseed quality using simple NIR instrument.

MATERIALS AND METHODS

The following samples of winter rapeseed were used for the investigations:

- 22 commercial samples, containing known amount of useless impurities, obtained from two oilseed plants,
- 18 breeding seed samples including traditional and impro-

ved varieties both, low erucic acid and low glucosinolates ("0" and "00"), obtained from three experimental stations.

Chemical analysis

The samples were analyzed by standard procedures in at least duplicate for:

- moisture content using air oven method,
- total protein (Nx6.25) by the standard Kjeldahl procedure,
- oil by the Soxhlet method using petroleum ether,
- glucosinolates according to Van Megen (1983) method,
- fatty acid composition by conversion of oils to methyl esters and the separation of esters in a 2 m long glass column packed with GP 10% SP-216-PS on 100/120 Supelcoport using a Chromatron GCHF-18.3 gas chromatograph.

NIR procedure

The samples for NIR analysis were prepared by grinding of rapeseed for 15 s in a high-speed coffee grinder. Well mixed samples were divided into sets of calibration and prediction and then were transfered into sample caps. For estimation of moisture, protein and oil contents in rapeseed, whole (intact) seed samples were also paralelly prepared. NIR analysis in triplicate was conducted with a Hungarian Infrapid 31 Analyzer.

RESULTS AND DISCUSSION

Since the calibration of instrument is critically important in the NIR technique, samples of rapeseed with wide range of variability in composition were chosen. Thus cleaned breeding seed samples and commercial samples (0.5-5.0% of useless impurities) were used. The moisture content in the samples varied from 4 to 17%, protein from 18 to 24%, oil 38-44%, erucic acid 0.5-52.0% and glucosinolates 2-18 mg/g of defatted dry matter. Statistical data (Tab.1) on rapeseed basic chemical composition have revealed a significant correlation between results obtained by standard

Table 1. Correlation between NIR and chemical analysis of rapeseed

Constituent	r[a]	y = ax + b	SDD[b]
	ground	samples	
moisture	0.9922**	y = 0.95x + 0.32	0.40
protein	0.9010**	y = 0.89x + 2.52	0.66
oil	0.8915**	y = 0.80x + 8.34	0.57
	intact	samples	
moisture	0.9295**	y = 0.86x + 1.02	1.13
protein	0.7055**	y = 0.58x + 8.87	1.07
oil	0.6552**	y = 0.56x +17.81	0.98

[a] Coefficient of correlation

[b] Standard deviation of differences

** Significant at $P \leq 0.01$

methods and NIR analysis using Infrapid 31 Analyzer. The highest correlation coefficient was estimated for moisture determination. Moisture is usually the most accurate measurement for NIR since it is the highest signal-to-noise ratio in the spectra (Robertson and Barton, 1984). Coefficients of correlation, especially for protein determination have been lower as compared to those of cereals which were obtained using the same instrument (Sałek and Deluga, 1984).

When intact seed samples were tested, lower correlation coefficients between standard methods and NIR results have been found (Tab.1). Moreover, differences in moisture, protein and oil contents between two compared methods were higher and standard deviation of differences increased about two fold, as well as precision of determination was diminished by 0.09% for moisture, 0.17% for protein and 0.16%

for oil (Tab.2). However, statistical evaluation of the results has proved that in all cases mean values of NIR vs standard methods were not significantly different ($P \leq 0.05$). This indicates that Infrapid 31 Analyzer can be considered as the instrument for routine analyses of basic chemical composition of the seeds when lower precision and accuracy could be accepted.

Table 2. Variability of basic composition in a single rapeseed sample by Infrapid 31 Analyzer

Sample	Moisture mean	Moisture $2S_{oi}$ [a]	Protein mean	Protein $2S_{oi}$	Oil mean	Oil $2S_{oi}$
ground[b]	4.8	0.26	21.7	0.26	41.5	0.15
intact[c]	4.8	0.35	21.9	0.43	41.2	0.31

[a] Precision

[b,c] Results of 12 and 8 analysis, respectively

Since in recent years improved rapeseed varieties have been introduced, it has become an important problem to control not only basic chemical composition but also such parameters as contents of erucic acid, glucosinolates. Our preliminary measurements suggest that Infrapid 31 Analyzer can be used for determination of glucosinolates in rapeseed. It is justified by high value of correlation coefficient of r=0.9379 (Tab.3), as well as lack of a significant difference ($P \leq 0.05$) of average values between two compared methods. Conducted measurements of erucic acid content by NIR have also shown significant correlation with the results of GLC method (Tab.3). However, great variability of the results (SDD=12.08) is unacceptable and indicates that NIR measurements using Infrapid 31 Analyzer are subjected to large error. No significant correlation ($P \leq 0.05$) between the NIR and standard methods for deter-

mination of linolenic acid and useless impurities contents has been found. However, it should be underlined that the results presented here are preliminary and further measurements on large sample population, also in respect to glucosinolates and erucic acid, are necessary.

Table 3. Calibration of rapeseed for percentage of glucosinolates and other constituents

Constituent	n	$\bar{d}_i$ [a]	SDD [b]	r [c]
glucosinolates	12	-0.82	2.96	0.9379**
erucic acid	9	-2.63	12.08	0.8025**
linolenic acid	9	-0.01	2.97	0.3613
useless impurities	18	-0.08	1.16	0.4116

[a] Average differences between NIR and standard procedure

[b] Standard deviation of differences

[c] Coefficient of correlation

** Significant at $P \leq 0.01$

REFERENCES

Brith G.S. and H.H.Ramey, 1982. Near-infrared reflectance for analysis of cottonseed for gossypol. Cereal Chem., 59:516-519

Kaffka K.J., K.H.Norris, J.Peredi and A.Balogh, 1982. Attempts to determine oil, protein, water and fiber content in sunflower seed by the NIR technique. Acta Alim., 11:253-269.

Robertson J.A. and F.E.Barton, 1984. Oil and water analysis of sunflower seed by near-infrared reflectance spectroscopy. J. Am. Oil Chem. Soc., 61:543-547.

Sałek M. and E.Deluga, 1984. Oznaczanie składu chemicznego ziarna zbóż metodą spektroskopii w bliskiej podczerwieni.

Proc. XV Sesja Naukowa KTiChŻ PAN Warszawa, A:1-3.

Van Megen W.H., 1983. Removal of glucosinolates from defatted rapeseed meal by extraction with aqueous ethanol. Can. Inst. Food Sci. Technol. J., 16:93-96.

Williams P.C., 1975. Application of near infrared refectance spectroscopy to analysis of cereal grains and oilseeds. Cereal Chem., 52:561-569.

Williams P.C., K.R.Preston, K.H.Norris and P.M.Starkey, 1984. Determination of amino acids in wheat and barley by near infrared reflectance spectroscopy. J. Food Sci., 49:17-20.