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THE EFFECT OF GRINDING EXTENT ON NEAR INFRARED SPECTROMETRY (NIRS) ANALYSIS OF SOME ANIMAL FEEDS

Sławomir Walczyński

National Research Institute of Animal Production
National Laboratory for Feedingstuffs in Lublin

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K e y w o r d s: NIRS technique, feedingstuffs, prepare a sample, grinding.

A b s t r a c t

A study was performed aimed at evaluating the method of sample grinding and its effect on the results of the analysis using the method of near infrared spectrometry. The object of the analysis was feed materials commonly used in animal feed manufacturing, such as cereal grain, wheat bran and high-protein materials (soybean meal). We also tested feed mixtures for poultry and pigs. Laboratory samples were ground on three milling devices – with the sieve of 1.0 mm or 0.5 mm, and a disc mill with minimum working aperture. The content of nutrients was analyzed with the help of the InfraXact spectrophotometer scanning within the range of 770–2,500 nm. The analyses revealed that within the range applied the method is resistant to the diversified granulometric composition of the samples. However, excessive grinding of the research material should be avoided (a sieve of 0.5 mm) during analyzing water, due to potential overheating of the sample which could lead to partial evaporation of the component.

Introduction

The nutritional value of animal feeds may be determined by specifying their chemical composition. However, standard analytical methods are time consuming and may generate high costs. An alternative for accredited methods could be near infrared spectrometry (NIRS) which is one of physical analytical methods. It reduces both the time and the costs of the analyses. The results obtained with the use of this particular method are reliable, accurate and may be archived (ANDERSEN et al. 2013, BUHŁAK 2006, KOLBUSZEWSKI 2009).

* Correspondence: Sławomir Walczyński, Krajowe Laboratorium Pasz, Instytut Zootechniki Państwowy Instytut Badawczy, ul. Chmielna 2, 20-079 Lublin, phone: 48 81 532 80 04 w. 117, e-mail: swalczynski@clpp.lublin.pl

The method of measurements made with a spectrometer consists of exposing a sample to electromagnetic radiation in the range of 770 nm to 2,500 nm. Due to the fact that the radiation is of low-energy type, it does not cause any alternations. The absorption of the radiation induces vibrations of chemical bonds in the compounds. These vibrations modify the initial signal, so the signal reaching the detector carries the information on any chemical compounds present in the sample. Since each type of a bond has a different characteristic point, the software used by the apparatus enables the researcher to identify particular compounds and to assess their quantity. The spectrum of the analyzed sample is compared with the mathematical model created during calibration and this provides the basis for predicting the parameters of the sample (BLANCO, VILLARROYA 2002, DEMSKI 2010, WANG, PALIWAL 2007).

All issues related to the use of near infrared spectrometry for determining certain parameters of feeding stuffs can be found in EN ISO 12099:2010 International Standard on Animal feeding stuffs, cereals and milled cereal products – Guidelines for the application of near infrared spectrometry. Its translation into Polish, authorized by the Polish Committee for Standardization, was first published at the beginning of 2013. Item 4 of the quoted standard presents the guidelines concerning measurement facilities, including milling or fragmenting devices used to prepare a sample. A comment accompanying this item includes a statement concerning a possible effect of fragmenting on the results of NIR. The present work discusses the results of using InraXact spectrometer to analyze samples which were prepared on three milling devices of different milling degree (DORSZEWSKI et al. 2004, PODKÓWKA, KOWALISZYN 2010).

The aim of the study

The present study aimed at assessing the influence of the degree of sample grounding on the results of the analysis performed with the use of near infrared spectrometry.

Material and methods

The analyses were performed on typical feeding stuffs: wheat, barley, maize grains, wheat bran and extruded soybean meal, as well as on powdered complete diets: starter, grower for poultry and finisher for pigs for fattening. Laboratory samples (one sample for each feedingstuffs) weighing 100 g were ground in:

1. Ultra Centrifugal Mill Retsch ZM 200 with ø 1.0 mm sieve,
2. Lab Mill 3100 FOSS with ø 0.5 mm sieve,
3. Cemotec Mill 9010 FOSS with minimum aperture.

The measurement of the granulometric composition in the ground samples was performed by means of the optical analytical method with the use of IPS UA analyzer, a device for specifying the granulation of solid particles in the air. In the optical measuring instrument the stream of infrared radiation was scattered by passed particles. After measuring a set of particles was converted to class dimensional (KAMIŃSKI et al. 2008). Each sample weighing ca. 4 g was measured three times. The anticipated content of moisture, crude protein, crude fat and ash was measured with the help of InfraXact, near infrared analyzer from FOSS, scanning within the wavelength range of 570–1,850 nm. The apparatus contained calibration set developed by FOSS, which are updated by RINA network (Remote Internet Analysis). Each sample weighing 10 g was scanned ten times. The basic validation parameter was determined, namely laboratory repeatability or the absolute difference between individual results obtained by means of the same method, performed on identical materials, in the same laboratory and by the same operator using the same equipment during a short time span. The measure of repeatability is the coefficient of result variation calculated according to the formulae 1–3.

Calculation of the mean value for a component from a series of 10 measurements, following formula (1):

$$\bar{x} = \sum_{i=1}^{n} \frac{x_i}{n} \tag{1}$$

x_i – value of each consecutive determination of the incremental sample,
$\bar{x}$ – the mean value of the determinations,
n – the number of determinations

and standard deviation, SD, from a series of 10 measurements, following formula (2):

$$\mathrm{SD} = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2} \tag{2}$$

SD – standard deviation.

Calculation of the coefficient of result variation, CV, following formula (3):

$$\mathrm{CV} = \frac{\mathrm{SD}}{\bar{x}}\, 100\% \tag{3}$$

CV – coefficient of variation.

Statistical analyses were performed with the use of Statistica package from StatSoft.

The results and the discussion

Sample results of analyzing the granulometric composition of the fragmented samples are presented in Figures 1–2 and in Table 1.

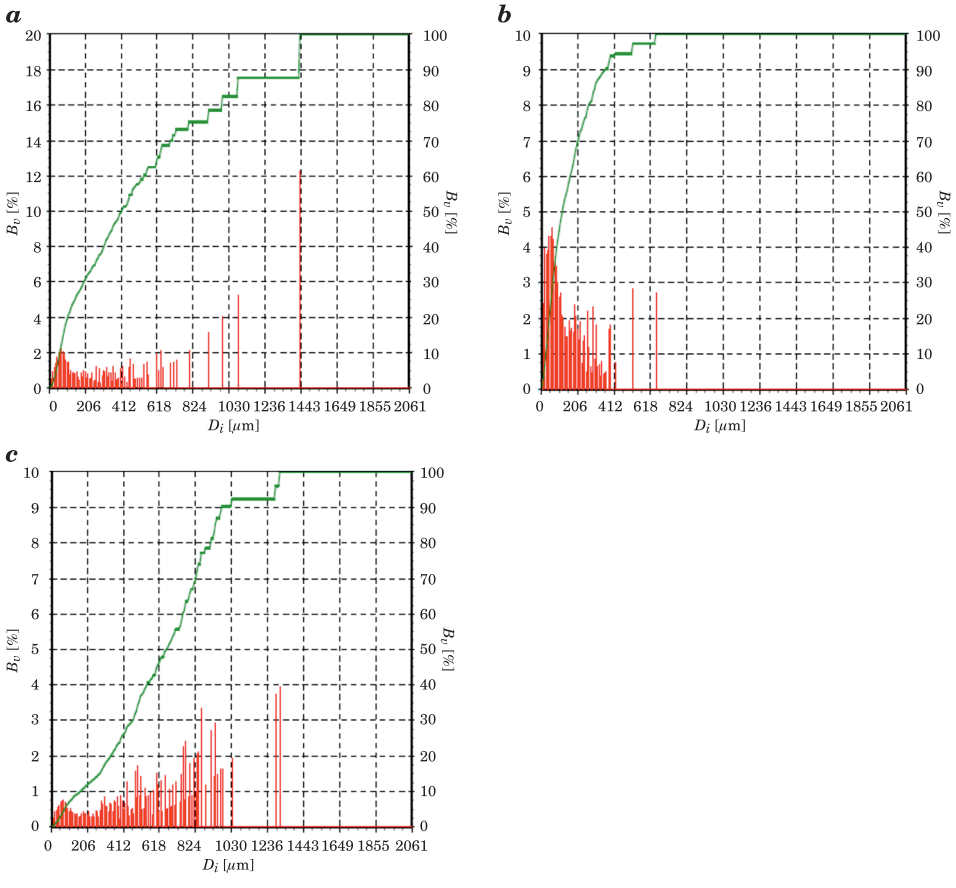

Fig. 1. Particle distribution in the samples of ground wheat: *a* – 1.0 mm sieve, *b* – 0.5 mm sieve, *c* – min. aperture

The smaller the sieve mesh in the grinder, the higher was the percentage content of fine particles in the milled feeding stuffs (Fig. 1, 2). The number of particles up to 200 μm ranged:

- for 1.0 mm sieve from 17.2% (grower diet for pigs) to 29.7% (wheat),
- for 0.5 mm sieve from 52.5% (grower diet for pigs) to 67.6% (wheat),
- for min. aperture from 11.5% (wheat) to 12.9% (grower diet for poultry).

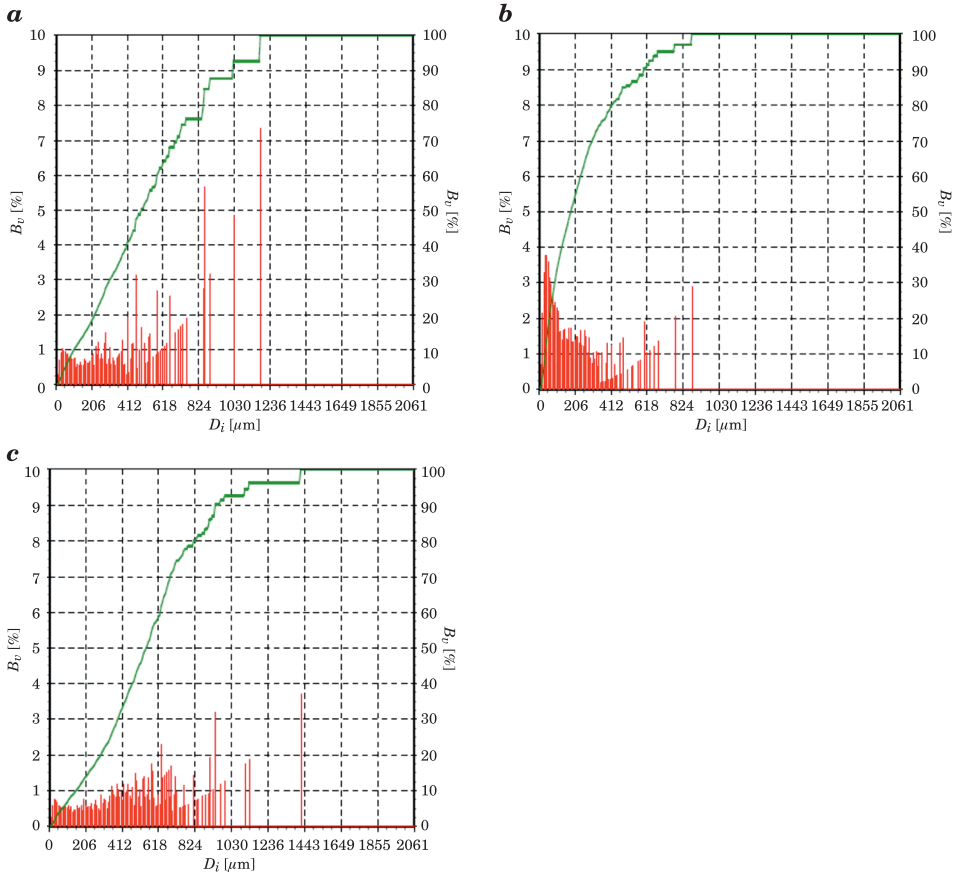

Fig. 2. Particle distribution in the samples of ground grower diet for pigs for fattening: *a* – 1.0 mm sieve, *b* – 0.5 mm sieve, *c* – min. aperture

The software used in the IPS UA analyzer let us calculate some characterizing parameters, such as the mean size of particles in the volume distribution, D_v, the diameter of particles marking exactly 50% of of the quantitative distribution, $D_{\text{mod.}}$ The obtained values were the lowest in case of using a ø 0.5 mm sieve, and a ø 1,0 mm sieve. The highest values of the analyzed parameters were observed in case of using a grinder with the minimum aperture (Table 1).

The analysis of the content of nutrients in relation to the degree of grinding in the samples of the evaluated feed materials (Table 2) and diets did not reveal any unambiguous influence of the sample size in the studied range on the analyzed parameters: moisture, protein, fat, crude fiber and crude ash (Fig. 3). However, we observed a decrease in the content of water in the samples ground

Table 1

Selected characteristics of ground feeding stuffs

Material/Sieve [mm]	D_n [μm]	D_v [μm]	D_{mod} [μm]
Wheat			
1.0	21.2	56.3	420.1
0.5	16.7	34.5	121.5
Min. aperture	19.3	71.4	661.8
Starter diet for poultry			
1.0	34.0	81.9	424.9
0.5	31.0	55.2	152.5
Min. aperture	38.4	104.3	597.0
Grower diet for pigs			
1.0	23.2	68.8	459.8
0.5	24.8	48.7	185.3
Min. aperture	22.3	72.9	551.1

D_n – average particle size in a quantitative distribution, D_v – average particle size in a volume distribution, D_{mod} – mean value of the particles determining exactly 50% of the quantitative distribution

Table 2

The analysis of the content of nutrients in relation to the degree of sample grinding – feed materials (mean values)

Grinding/ sieve	Component	Wheat	Maize	Barley	Wheat bran	Soybean meal
1.0 mm	protein [%]	14.67^{a}	8.11^{a}	10.36^{ab}	17.22^{a}	48.04^{a}
0.5 mm	protein [%]	14.67^{a}	8.72^{b}	10.19^{b}	16.98^{b}	47.18^{b}
min. aperture	protein [%]	14.76^{a}	8.30^{c}	10.50^{a}	17.35^{a}	$47.80c$
1.0 mm	moisture [%]	11.60^{a}	12.11^{a}	11.75^{a}	13.55^{a}	$10.69a$
0.5 mm	moisture [%]	11.44^{b}	12.06^{a}	11.46^{b}	12.52^{b}	$10.29b$
min. aperture	moisture [%]	11.62^{a}	12.15^{a}	11.89^{a}	13.97^{c}	10.96^{c}
1.0 mm	fat [%]	1.58^{a}	4.01^{a}	1.97^{a}	3.93^{a}	1.18^{a}
0.5 mm	fat [%]	1.49^{b}	3.68^{b}	1.85^{b}	4.11^{b}	0.88^{b}
min. aperture	fat [%]	1.45^{b}	3.90^{a}	1.32^{c}	3.78^{c}	1.17^{a}
1.0 mm	fiber [%]	3.38^{a}	2.51^{a}	4.00^{a}	8.48^{a}	3.33^{a}
0.5 mm	fiber [%]	3.19^{b}	2.24^{b}	3.49^{b}	8.71^{b}	3.45^{b}
min. aperture	fiber [%]	2.95^{c}	1.97^{c}	3.42^{b}	8.39^{a}	3.19^{c}
1.0 mm	Ash [%]	1.72^{a}	1.28^{a}	2.00^{a}	4.41^{a}	6.53^{a}
0.5 mm	Ash [%]	1.62^{b}	1.24^{b}	2.04^{a}	4.50^{a}	6.56^{b}
min. aperture	Ash [%]	1.60^{b}	1.19^{c}	1.83^{b}	4.07^{b}	6.47^{c}

a, b, c – significant differences, with $p < 0.05$, within component and feed material

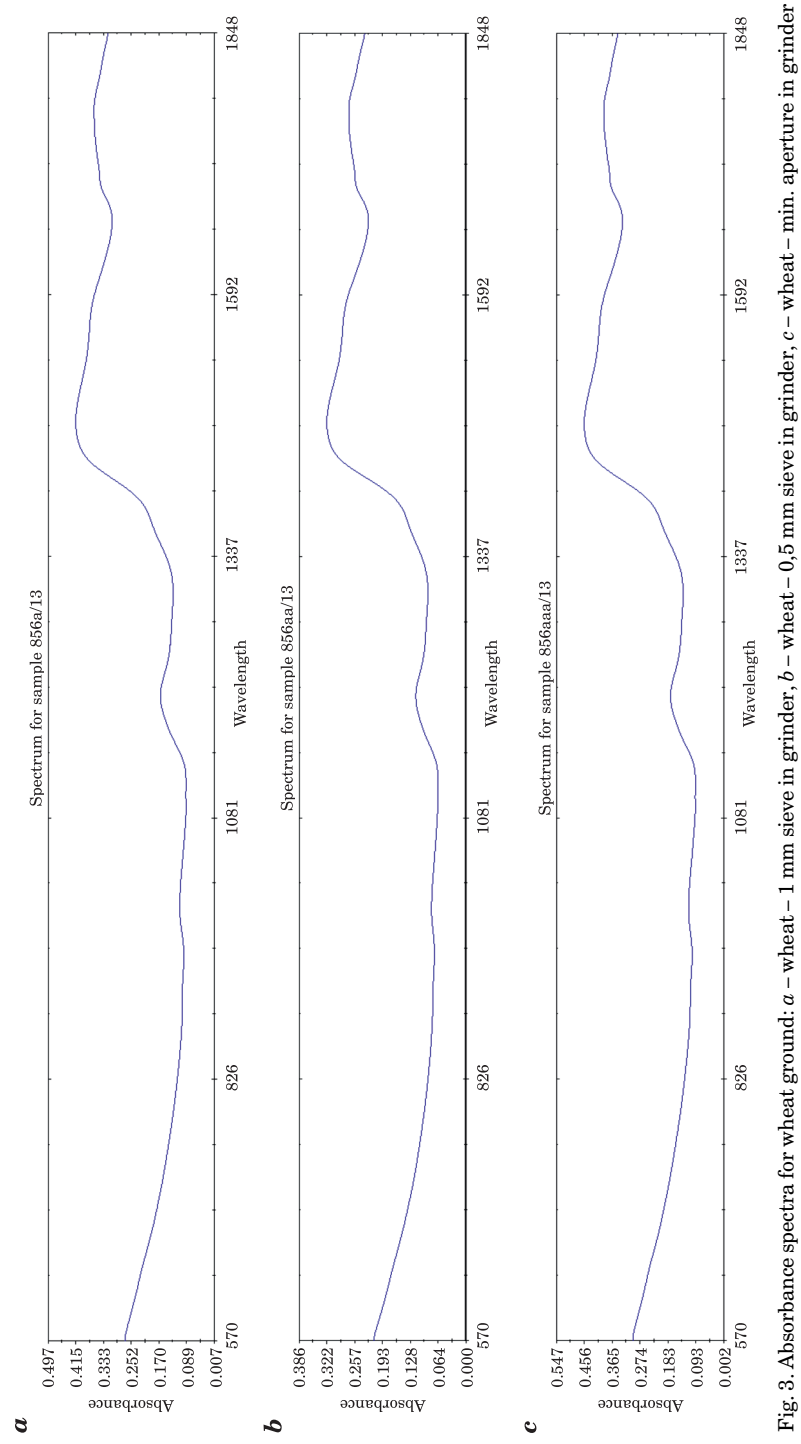

Fig. 3. Absorbance spectra for wheat ground: *a* – wheat – 1 mm sieve in grinder, *b* – wheat – 0,5 mm sieve in grinder, *c* – wheat – min. aperture in grinder

Table 3
Laboratory repeatability in relation to the degree of sample grinding – feed materials

Material	Parameter/Value determined [%]	Repeatability [%]		
		grinding		
		1.0 mm	0.5 mm	min. aperture
Wheat	protein – 14.7	1.10	0.64	0.92
Maize	protein – 8.38	1.97	1.05	1.23
Barley	protein – 10.35	1.98	2.41	1.50
Wheat bran	protein – 17.18	0.93	0.78	0.88
Soybean meal	protein – 47.67	0.27	0.27	0.42
	Mean value	**1.25**	**1.03**	**0.99**
Wheat	moisture – 11.55	0.82	1.13	0.88
Maize	moisture – 12.11	1.09	0.95	1.48
Barley	moisture – 11.17	0.62	1.73	1.58
Wheat bran	moisture – 13.45	0.91	2.15	1.80
Soybean meal	moisture – 10.65	2.22	0.56	0.94
	Mean value	**1.13**	**1.30**	**1.34**
Wheat	fat – 1.57	3.89	4.63	5.02
Maize	fat – 3.83	2.94	1.13	7.37
Barley	fat – 1.71	6.12	3.06	7.50
Wheat bran	fat – 3.94	2.25	2.77	1.65
Soybean meal	fat – 1.08	5.20	7.34	4.46
	Mean value	**4.08**	**3.79**	**5.20**
Wheat	fiber – 3.27	3.64	4.63	5.02
Maize	fiber – 2.24	2.85	4.65	7.51
Barley	fiber – 3.64	4.12	3.61	4.60
Wheat bran	fiber – 8.53	1.92	1.36	1.75
Soybean meal	fiber – 3.32	2.60	2.54	2.05
	Mean value	3.03	3.36	4.19
Wheat	ash – 1.65	2.87	1.35	1.85
Maize	ash – 0.90	0.78	1.83	3.33
Barley	ash – 1.96	2.25	2.81	1.33
Wheat bran	ash – 4.32	4.14	3.32	7.57
Soybean meal	ash – 6.52	0.36	0.55	0.32
	Mean value	**2.08**	**1.97**	**2.88**

with the use of the ø 0.5 mm sieve, as compared to the other methods of milling. During processing the sample was heated, so some of the water could have evaporated. The content of fiber and crude ash was lower in the samples ground on the disc mill. However, the differences were not statistically significant in some cases.

Table 3 presents a comparison of the values calculated for laboratory repeatability, depending on the degree of sample grinding for feed materials. Similar results were obtained for feed diets. The values in particular groups did not differ significantly. Average laboratory repeatability for feed materials and the sieve used were, respectively, 1.0 mm sieve – 2.31%, 0.5 mm sieve – 2.29%, min. aperture – 2.92%. This parameter was comparable with the data obtained in validation examinations performed in the National Feed Laboratory in Lublin (Table 4).

Table 4

Laboratory repeatability in validation examinations in the National Feed Laboratory, following the Commission Regulation No 152/2009 of 27 Jan. 2009

Analysis	Laboratory repeatability [%]
Protein	1.0
Moisture	1.1 – 1.4
Fat	1.4 – 1.5
Fiber	2.8 – 4.25
Ash	1.3 – 2.0

Summary

1. Within the range of the studied degree of grinding the method of analyzing the content of nutrients with the use of the InfraXact apparatus is low sensitive to the diversified granulometric composition of the samples.

2. However, while determining humidity excessive degree of sample grinding should be avoided (0.5 mm sieve), since the material may become overheated and some water could evaporate.

3. Laboratory repeatability for the results obtained by means of the NIRS method is comparable with repeatability of the accredited methods following Commission Regulation (EEC) No 152/2009.

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