Analysis of mixed feeds and their components using near infrared spectroscopy

Peter Tillmann, a Hartmut Horst, b Jürgen Danier, c Peter Dieterle d and Petra Philipps e

^aVDLUFA, Am Versuchsfeld 13, D-34128 Kassel, Germany

^bHLDGN, Am Versuchsfeld 13, D-34128 Kassel, Germany

^cHVA, D-85350 Freising, Germany

^dLUFA Rheinland-Pfalz, Obere Langgasse 40, D-67346 Speyer, Germany

^eUntersuchungszentrum Bonn-Roleber LUFA, Siebengebirgsstr. 200, D-53229 Bonn, Germany

Introduction

Mixed feeds and their components are a very diverse matrix compared with other agricultural products where near infrared (NIR) spectroscopy has classically been applied. On a database of mixed feeds and their components (n = 2.130), universal partial least squares (PLS) calibrations were developed. The results from validation (five sets with n = 180, 130, 124, 41 and 96) show the potential of the calibrations and their limitations. Crude protein, crude fibre, crude fat, starch and sugar were predicted on a dry matter basis with standard errors of prediction (SEP) of 1.0%, 1.0%, 0.5%, 1.5–2.0% and 1%, respectively. Gas production was predicted with SEP of 2–2.5 mL for a sample of 0.2 g (dry matter basis). Ash content of 15% and more in several mixed feeds or components, as well as rare components limit the use of NIR for routine analyses.

The objective was to show the potential and limitations of using NIR for a heterogeneous matrix as mixed feeds and their components.

Material and methods

Over a three-year period, feed samples were collected from traded feeds (mixed feeds and raw components). On the basis of preliminary results, pet food and feeds with an ash content above 15% (for example, fish meal and laying hen feeds) were excluded from the study. The data set consisted of 2130 samples, from which three validation sets were separated (all samples from 1998: (n = 180), a random selection of samples from 1999 (n = 130) and 2000 (n = 124). The characteristics of samples in the calibration set are shown in Table 1. Furthermore, two additional data sets consisting of mixed cow feeds (n = 41) and mainly pig feeds (n = 96) were collected in different regions in Germany,

All samples were ground and were scanned on an NIRSystems 5000 (Foss, Denmark). Calibrations were developed using ISI software¹ with standard settings (modified partial least squares, multiplicative scatter correction, 1,4,4,1, cross-validation). Reference analyses were done according to

Table 1. Samples in the calibration set.

	Number
Grains	317
Concentrates for cows	242
Pig feed	222
Oilseed meals, cakes	183
Piglet feed	143
Concentrates for fattening cattle	110
Legumes	107
Concentrates for horse	99
Milling by-products	80
Calve feed	71
Concentrates for sheep	39
Poultry feed	33
Others	42

VDLUFA (1997)² for crude protein (XP, Kjeldahl), crude fibre (XF, Weende analysis), crude fat (XL, Soxhlet extraction), starch (XS, Ewers), sugar (XZ, Luff-Schoorl) and gas production (GP)³ in three different laboratories. The unbalanced distribution in the calibration set is shown in Table 2. Due to the targeted use of the samples, not all constituents were analysed on all samples, i.e. starch and sugar were carried out mainly on pig feeds while gas production was measured only on concentrates for cows.

Results and discussion

The calibrations allow the determination, on a dry matter basis, of crude protein (XP), crude fibre (XF) and sugar (XZ) with *SEP*s of approximately 1.0%. Crude fat (XL) was predicted with an *SEP* of 0.5%, starch (XS) with an *SEP* of 1.5–2.0%, while gas production (GP) can be estimated with an *SEP* of 2–2.5 mL per 0.2 g (see Table 3).

Table 2. Distribution of calibraiton samples.

Constituent	n	Mean	Mean Range	
XP	1635	20.6	3.9-64.9	10.9
XF	1051	8.7	0.3–32.5	4.3
XL	748	4.2	1.0-26.3	2.0
XS	692	43.2	0.8–79.9	19.8
XZ	452	6.9	0.7–31.5	3.4
GP	253	56.0	32.5–75.3	6.9

Table 3. Validation of the mixed feed calibration with five validation sets (SEP).

Data set/ Constituent	1998	1999	2000	R-P	NRW
XP	1.42	0.98	1.00	0.89	1.1
XF	1.21	0.63	1.22	1.14	0.9
XL	0.54	0.45	0.59	0.41	0.3
XS	1.44	1.36	1.60	_	2.2
XZ	0.87	0.98	0.62	_	0.9
GP	3.23	2.50	2.11	1.79	_

P. Tillmann et al.

Table 3(a). Composition of validation sets.

Data Set	1998	1999	2000	R–P	NRW	
Concentrates						
Cows	41	24	22	41	2	
Fattening cattle	17	15	11	_	10	
Sheep	18	2	2	_	_	
Horse	14	13	15	_	_	
Other types						
Calf feed	9	10	10	_	_	
Piglet feed	19	18	19	_	1	
Pig Feed	24	23	20	_	64	
Poultry feed	_	_	_	_	11	
Soybean & other oil seed meals	14	4	5	_	_	
Milling by-products	14	11	16	_	_	
Miscellaneous	10	10	4	_	8	

The results for crude protein and crude fat compare favourably with results reported by Berzaghi *et al.*⁴ for PLS calibrations for feeds. The best LOCAL procedure (*SEP* 0.8 for XP and 0.4 for XL) reported in the same study were not matched in this study.

Cizmar⁵ reported *SEP* for mixed pig and poultry feeds of 0.5%, 0.6%, 0.3% and 1.4% for crude protein, crude fibre, crude fat and starch, respectively. In the present study, samples were collected from mixed feeds as well as raw materials. In the validation sets, about 20% of the samples were raw products.

Conclusions

These calibrations permit the estimation of energy values for the production of mixed feeds and for trading feeds. The NEL of mixed cow feed can be estimated with an *SEP* of 0.15 MJ. For pig feed, the energy estimation has a *SEP* of 0.4 MJ ME, due to the increased *SEP* of starch estimation. All conclusions are based on the official German energy estimation scheme.⁶

References

- 1. ISI, WinISI II. Software manual, Version 1.00. Infrasoft International, LLC (1998).
- 2. VDLUFA, Methodenbuch Band III. Die chemische Untersuchung von Futtermitteln. VDLUFA-Verlag, Darmstadt, Germany (1997).
- 3. K.H. Menke, L. Raab, A. Salewski, H. Steingass, D. Fritz and W. Schneider, *J. Agric. Sci. Camb.* **93**, 217 (1979).
- 4. P. Berzaghi, J.S. Shenk and M.O. Westerhaus, J. Near Infrared Spectrosc. 8(1), (2000).
- 5. D. Cizmar, oral presentation, 29–30 May, ALVA Tagung, Wolfpassing, Austria (2001).
- 6. GfE, Proc. Soc. Nutr. Physiol. 5, 53 (1996).