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APPLICATION OF FT-NIR SPECTROSCOPY FOR EVALUATION OF FEEDS DIGESTIBILITY BY ANALYSIS OF FECES CHEMICAL COMPOSITION

Semyon BIBIKOV, Sergii SHAPOVALOV, Elena KORNILOVA

Abstract. In this study, we demonstrated the possibility of rapid and non-destructive quantification of crude protein, fat, fiber and moisture content in pigs' feces by Fourier transform near-infrared (FT-NIR) spectroscopy. The predictions of developed partial least-squares (PLS) regression calibration models do not exceed the error of used arbitrary method. The FT-NIR models demonstrate satisfactory correlation coefficients (R^2 >0.81) and residual predictive deviation (RPD) values from 2.3 to 7.5, depending on measured parameter. Obtained results indicate the possibility of use FT-NIR spectroscopy as a simple tool for monitoring feed's digestibility and allowing applying timely corrections to diets.

Key words: Pigs; Feces; Feed digestibility; Infrared spectroscopy.

INTRODUCTION

Over past decades, the question of measuring the nutritional value of feed for farm animals and poultry receives more and more attention among nutritionists (Han, S. et al. 2015; Shurson, G. C. 2015; Blair R. 2017; Bourgon, S. et al. 2018). At the same time, the widespread use of computing and the development of multidimensional statistical procedures in the field of chemometrics led to the widespread use of infrared spectroscopy (FT-NIR). This technique became a good alternative to traditional analytical methods for determining the nutritional value of feed (Danezis, G.P. et al. 2016; Danezis, G.P. et al. 2016; Ingle, P.D. et al. 2016).

The decision in time on the adjustment of farm animals' diet improves the overall profitability of production. Numerous studies showed the applicability of FT-NIR to analyze not only raw materials for feed production, but also the products of nutrient digestion/fermentation (Stuth, J. A. et al. 2003; Althaus, B. G. 2013; Fredin, S. et al. 2014; Tolleson, D. et al. 2014; Pierre-Olivier, J. et al. 2015). In other words, the method of infrared spectroscopy makes it possible to control the quality of feeding animals through the analysis of their feces.

In early 1990s, the research group of Stuth J. W. (1992) used the infrared spectroscopy for assessing the quality of feeding cattle on grazing by analysis of animals' feces. The measured parameters included the content of crude protein and organic matter. The obtained IR spectra were used for the development of a predictive equation by stepwise regression. Standard calibration error (SEC) and standard error of validation (SEV) for organic matter were 1.66 and 1.65 respectively, although they did not exceed the laboratory standard error (SEL) of 1.68. SEC and SEV parameters for crude protein were 0.89 and 0.93, respectively, compared with 0.44 SEL. In a later work (Leite, E.R., Stuth, J.W. 1994), the same author conducted a similar study, choosing goats as a model animal.

L. J. Jancewicz et al. in 2016 presented a more complete chemometric model based on investigation of cattle feces, allowing spectroscopic determination of dry and organic matter, starch, nitrogen, neutral detergent fiber (NDF), acidic -detergent fiber (ADF), acid-detergent lignin (ADL) and fat. The primary model showed a significant correlation (R^2> 0.70) for organic matter, starch, nitrogen, NDF and ADF, while for ADL it was significantly lower (R^2 <0.25). The expansion of the calibration spectra set with new samples made it possible to improve the accuracy of predictions for all components of feces (R^2>0.90; SECV<2.42). The accuracy of prediction of digestibility was high for starch (R^2=0.84, SECV=1.06), moderate for dry and organic matter and crude protein (R2≥0.62, SECV≤3.63), but poor for NDF and ADF. (R^2≤0.33, SECV≥7.86). J. R. Johnson in 2017 obtained similar data. K. G. Nirea in 2018 conducted research on pigs, showing that infrared spectroscopy is a cost-effective method for determining the digestibility in conditions of intensive animal husbandry.

Thus, infrared spectroscopy is a promising technique for the analysis of animal feces, combining the simplicity of use and speed of the result. The aim of our research was to develop chemometric models

for assessing the digestibility of feed, determined on chemical composition of pigs' feces. The chosen model parameters included moisture, crude protein, crude fat and crude fiber.

MATERIALS AND METHODS

The studies were conducted on the basis of "Kuznetsovsky Combinat" CJSC and Research and Testing Center "Cherkizovo" LLC., which are part of "Cherkizovo" PJSC. More than 60 samples of swine feces from the farm (Fig. 1) were analyzed.

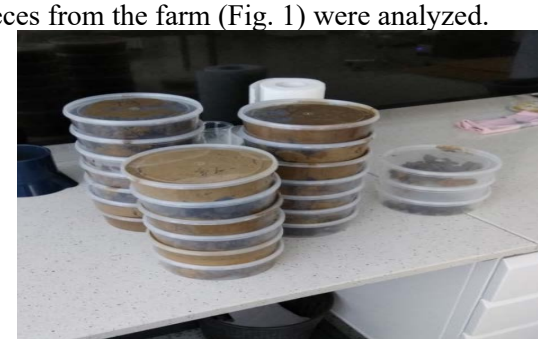

Figure 1. *The samples of swine feces*

Figure 2. *The samples of swine feces in a Petri dish*

The feces were studied using the following arbitration techniques:

- moisture by drying the sample to constant weight at a temperature of 103±2 °C according to GOST R 54951-2012;
- crude fat by extraction of the sample in the Soxhlet apparatus according to GOST 13496.15-2016;
- Crude protein by the method of determining the total nitrogen according to the Kjeldahl method according to GOST 13496.4-93, followed by conversion to protein;
- crude fiber content by using the method of intermediate filtration according to GOST ISO 6865-2015.

To record the spectra, the FT-NIR spectrometer MPA (Bruker, Germany) was used. The measurements were carried out in the integrating sphere mode, by averaging of 3 recorded spectra in order to eliminate the possible heterogeneity of feces composition. Measurement parameters were used as follows: resolution - 16 cm^{-1}; the wave number range is 12,400 - 3,600 cm^{-1}. All the samples were investigated "as is" without any pretreatment (Fig. 2).

All the measurements were carried out in accordance with the user's guide of the FT-NIR spectrometer (ASTM E 1655-05-2005). The survey spectra of feces are presented in Figure 3.

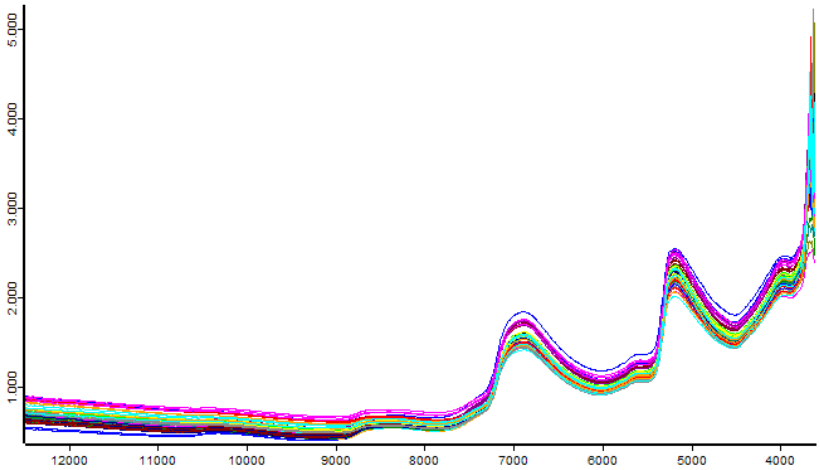

Figure 3. *The survey spectra of swine feces in the range 12400 – 3600 cm^{-1}*

Calibration model was obtained using OPUS QUANT software. The program implements the Partial Least Square Regression (PLS) method (Wold, S. et al. 1983), based on a nonlinear repetitive prediction algorithm for changing least squares. In the Russian-language literature, this method is better known as the "method of projection on latent structures".

The quality of the calibration model obtained was evaluated according to the following criteria:

- the data are within the 95% confidence interval,
- uniform coverage with points (samples) of the entire calibration working range;
- all points are arbitrarily located near the line y = 0;
- the number of points far from the main body, or the number of "emissions" does not exceed 5%;
- the RMS cross-validation error (RMSECV) or RMS estimation error (RMSEE) must be within the reproducibility of the standard method.

The calculation of the error is carried out automatically with the subsequent displaying the results.

The calibration model was tested on an independent sample set. Samples for testing were not included in the main set of calibration samples. The values for moisture, crude protein, crude fat and crude fiber in the test samples were determined by wet chemistry methods. The preparation and analysis of test samples was carried out similar to samples used for calibration.

RESULTS AND DISCUSSION

In total 60 samples were used to develop the calibration model, based on the spectral data and ones of "wet" chemistry (Figure 4). The quality of developed chemometric model was additionally tested in terms of the absence of false positive / negative response during the analysis of random samples (Table 1).

Table 1. *Main parameters of the developed calibration model*

Component	Concentration range, %		R^2	RPD	RMSEP/ RMSEE
	min	max			
Moisture	65.90	84.10	0.98	7.48	0.67/0.61
Crude protein	3.80	10.27	0.90	3.16	0.48/0.44
Crude fat	1.34	3.77	0.82	2.39	0.31/0.28
Crude fiber	3.14	5.71	0.81	2.31	0.42/0.37

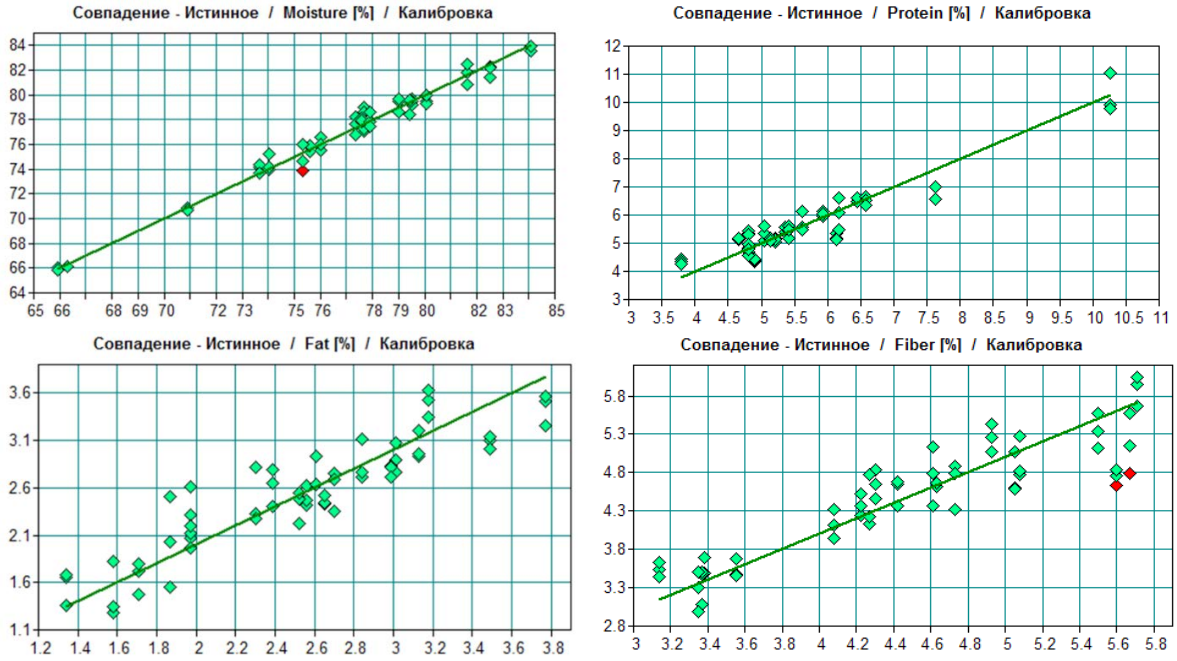

Figure 6. *Calibration models of various physicochemical parameters: a) moisture content, b) crude protein, c) crude fat, d) crude fiber*

To test the developed calibration model, 5 feces samples not included in the calibration set were used. The validation of the obtained model showed satisfactory the results for all parameters (Table 2).

The difference between the values obtained by the arbitrary methods and infrared spectroscopy did not exceed the reproducibility (R) of the arbitration method.

Table 2. *Comparison of values obtained by chemistry and FT-NIR model*

Sample		1	2	3	4	5
Water	IR	83.40	86.20	87.30	86.70	83.50
	Chem	83.00	86.70	87.10	86.50	84.00
	Δ	0.40	0.50	0.20	0.20	0.50
	R	-	-	-	-	-
Crude protein	IR	10.27	6.17	5.14	5.94	7.62
	Chem	10.81	5.67	5.54	5.51	7.95
	Δ	0.54	0.50	0.40	0.43	0.33
	R	0.69	0.53	0.49	0.52	0.58
Crude fat	IR	3.01	2.99	1.97	2.39	3.18
	Chem	3.43	3.43	1.77	2.02	3.58
	Δ	0.42	0.44	0.20	0.37	0.40
	R	0.52	0.52	0.47	0.49	0.53
Crude fiber	IR	3.40	4.60	5.60	4.90	5.70
	Chem	4.20	3.70	6.70	5.80	4.80
	Δ	0.80	0.90	1.10	0.90	0.90
	R	1.10	1.20	1.20	1.20	1.20

CONCLUSIONS

The obtained results proved that developed calibration model is stable and rigid. Thus, the FT-NIR technique allows rapid obtaining the data on chemical composition of swine feces. This information can be used in assessing the digestibility of particular animal feed. The validation of calibration equation by the independent set of samples demonstrated that errors of prediction are within the limits of the reproducibility of the arbitrary method.

The further expansion of the set of calibration spectra will increase the accuracy of prediction, as well as provide an opportunity to expand the number of determined physicochemical parameters, such as acid-detergent fiber (ADF), neutral detergent fiber (NDF), amino acids, starch, total sugars, energy etc.

The obtained results indicate the applicability of use FT-NIR spectroscopy not only for rapid analysis of swine feces, but also for analysis the physicochemical parameters in feces of other farm animals. This finding opens wide perspectives for use of FT-NIR technique for determining the quality of organic fertilizers made from animal feces.

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INFORMATION ABOUT AUTHORS

BIBIKOV Semyon Olegovich https://orcid.org/0000-0002-9747-4194

Senior specialist in the direction of testing feed and products of animal origin, Research and Testing Centre «Cherkizovo» Ltd., Moscow, Russian Federation

E-mail: bibikovso@gmail.com

SHAPOVALOV Sergii Olegovich https://orcid.org/0000-0001-7157-5627

Doctor of Biological Sciences, director of Research and Testing Centre «Cherkizovo» Ltd., Moscow, Russian Federation

E-mail: s.shapovalov@cherkizovo.com

KORNILOVA Elena Vyacheslavovna https://orcid.org/0000-0001-7852-4945

PhD in Agricultural Sciences, head of direction of testing feed and products of animal origin, Research and Testing Centre «Cherkizovo» Ltd., Moscow, Russian Federation

E-mail: e.kornilova@cherkizovo.com

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