

APPLICATION OF NIR TECHNOLOGY IN THE ANIMAL FOOD INDUSTRY

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Abstract: The importance of NIR technology in the animal food industry is presented in this study. As the example of the calibration procedure of NIR devices a calibration model for 14 samples of soybean cake was designed. Samples were previously analyzed in the standard laboratory testing of the moisture content, content of crude proteins, crude fats and crude fibre. In this calibration procedure high determination coefficients - R^2 were established for these parameters of the nutritional value of food (0.9783 for moisture, 0.9904 for crude proteins, 0.9872 for crude fats and 0.9351 for crude fibre). The comparison of values obtained by using standard laboratory methods with values obtained by NIR technology/method indicates that by using NIR devices it is possible to obtain highly reliable results, and therefore it can be used successfully in facilities for production of animal food in the control of the quality and projection of mixtures.

Key words: NIR, animal food

Introduction

Adequate nutrition of animals includes knowledge of the characteristics of used feeds (Jovanovic *et al.*, 2009). Also, intensive rearing of farm animals and stringent requirements in regard to the quality and quantity of animal products, require constant monitoring of the chemical composition of food for animals. On the other hand, strong competition in the field of production of animal food, imposes higher efficiency of the production process. Results obtained in the standard chemical analysis methods of major quality parameters of raw materials and finished mixtures are often received too late to stop the production process on time or correct it (Vries *et al.*, 2010). Therefore, it is necessary to use fast analytical methods which enable fast response in cases when certain deviations are observed from the projected composition or quality of the product. In this way it is possible to achieve significant savings in the production process and ensure product of stable quality. NIR method of analysis is exceptionally fast (usual duration of

analysis is 10 seconds), non-destructive and does not require preparation of the sample for analysis.

Main principle of NIR method of analysis. NIR(S) method (Near Infrared Reflectance Spectroscopy) is based on screening of sample by using near infrared reflectance/light, resulting in the spectrum of this individual sample. Spectrum can represent dependence of the reflected or transmitted radiation from the wave length. Results of the indirect NIR method, i.e. spectrum data, subsequently have to be transformed into required results – concentration of the relevant constituent or functional property of the tested material, which is obtained by developing/designing of the calibration model. Calibration of NIR devices is done by using samples of known composition and properties determined by standard (reference) analysis methods. Calibration procedure includes application of certain mathematical and statistical techniques (chemometrics) for the purpose of obtaining of empirical equation which connects spectrum data with data obtained by chemical analysis (*Stuth et al., 2003*). The costs associated with chemical analysis of large number of samples (minimum 50, whereas approx. 150 chemical analyses of different samples is required for open calibration sample population) are typical for single chemometric method. Calibration of NIR devices requires certain amount of time, but it is facilitated by the possibility of sophisticated software packages which provide for the user application of chemometric calibration techniques (MLR Multiple Linear Regression, PLS – Method of Partial Least Squares Regression, PCR – Principal Component Regression, ANN – Artificial Neural Network, etc). However, there are multiple benefits/advantages provided by the NIR method which are beyond the time and money invested in its implementation.

Development of calibration model represent key step in successful implementation of NIR method and it comprises following phases:

- Selection of calibration set of samples; robustness and accuracy of the calibration model greatly depend on the variability of the calibration population in sense of presence of samples of different varieties, samples of various maturity stages, samples originating from different cultivating regions and different production years (*Tsuchikawa, 2007*);

- Collection of spectrum and reference data;

- Execution of the regression (calibration) model and,

Validation of the model; objective of the calibration model validation is to assess its predicting abilities in a routine application (*Petersen, 2007; Boysworth and Booksh, 2008*). We distinguish two types of validation procedure of NIRS calibration model; cross-validation and external validation, i.e. validation using an independent set of samples.

Both procedures result in prediction error, i.e. error which can be expected in a routine application of model used to determine the quality, i.e. efficiency of the developed model (*Esbensen, 2006*), and which is based on the concept of

differences between NIR results and results of reference laboratory analyses (Isaksson and Segtnan, 2007; Shenk et al., 2008).

Materials and Methods

Samples of soybean cake were analyzed using standard chemical methods (reference methods) and NIR method, in order to compare results of these two methods. Analyses were done in FSH Komponenta from Čuprija. The analysis of the moisture content, content of crude fats and proteins was done on 14 samples, and analysis of crude fibre content on 9 samples of mentioned feed. All analyses were done according to the Rulebook on methods of physical, chemical and microbiological analysis of animal food from year 1987. Analysis of the moisture content was done on the laboratory moisture - meter OHAUS® type MB45 for fast determination of moisture content. Chemical analysis of crude proteins was done according to method by Kjeldahl; for determination of the crude fats content the procedure of dry extraction according to Soxhlet method was applied, using VELP® Scientifica digester and fat extraction apparatus. In chemical analysis of crude fibre the Weender method was used. Chemical analysis was done in two repetitions and average value of obtained results was considered in the study. For NIR analysis of samples NIR device was used - PERTEN Diode Array 7200, with rotating dish for measuring of samples, of diameter of 75 mm. This type of NIR device does not require previous grinding of samples, which is also an advantage. The wave length range from 950 to 1650 nm was used for measuring. The following mathematical transformations were carried out, on the spectrum of the soybean cake sample, in order to remove all irrelevant information from the spectrum, and to be able to interpret the results easier: Savitzky-Golay – to obtain first spectrum excerpt and MSC – Multiplicative scatter correction, and for the purpose of development of calibration model, PLS1 – Partial Least Squares Regression. For transformation of the main spectrum of the soybean cake samples and execution/development of calibration model software package CAMO Unscrambler® 10.1 was used.

Results and Discussion

In assessment of the efficiency of the obtained calibration model the following statistical tests were used: RMSEP – Root Mean Square Error of Prediction and R^2 – Determination coefficient R^2 (Table 1). Root Mean Square Error of Prediction – RMSEP represent measure of the variability of differences between values obtained by NIR analysis (predicted values) and reference laboratory methods. When the value of RMSEP is closer to the zero, the calibration model is more reliable. Determination coefficient R^2 , represents the square of the correlation coefficient – r , and describes the variation and range of the calibration

set. Value of $R^2 = 1$, means that 100% of variations are described with the calibration (Pojić, 2010). Based on presented results it can be concluded that very good determination coefficients (R^2) in all cases are obtained. In comparison of concentration values of tested parameters obtained by standard laboratory method and those obtained by NIR method (Table 2), it is observed that results of the NIR method of analysis show no significant deviations from results obtained by standard laboratory methods, and errors which occurred did not exceed deviation regulated in the Rulebook on quality and other requirements for animal food („Official Journal of RS“, no. 4/2010). The highest determination coefficient (R^2) was achieved in crude proteins, which was expected since N-H chemical bond present in proteins shows high level of absorption of infrared radiation (Oatway et al, 2006). Very good determination coefficient (R^2) was also achieved in crude fats, although the concentration range of this constituent was considerably wide. Slightly lower determination coefficients (R^2) were obtained from moisture and crude fibre. On the other hand, value of the Root Mean Square Error of Prediction - RMSEP was the lowest in crude fibre (lower than in crude proteins) which is explained by very narrow range of content of this parameter.

Table 1. Concentration of crude proteins and crude fats in the calibration set (%), range of their values and results of the statistical cross validation

	Average of values, %	SD	Range	R^2	RMSEP
Moisture	7.31	1.52	4.06 – 9.65	0.9783	0.2442
Crude proteins	38.12	0.53	37.29 - 39.15	0.9905	0.0517
Crude fats	7.95	1.21	6.49 - 11.31	0.9884	0.0972
Crude fibres	6.02	0.32	5.72 – 6.91	0.9351	0.04331

SD – Standard deviation, R^2 – Determination coefficient, RMSEP – Root Mean Square Error of Prediction.

Table 2. Comparative review of the results of standard laboratory and NIR methods of analysis

Sample	Moisture		Crude proteins		Crude fats		Crude fibres	
	St.l.m, %	NIR, %	St.l.m, %	NIR, %	St.l.m, %	NIR, %	St.l.m, %	NIR, %
1	4.060	4.446	37.780	38.669	8.880	8.761	5.720	5.742
2	7.540	7.752	38.140	38.202	7.000	7.131	6.080	6.001
3	7.000	6.849	37.720	37.674	8.880	8.985	5.900	5.927
4	8.080	8.373	37.810	37.816	7.180	7.082	5.880	5.882
5	8.160	8.047	37.400	37.391	7.290	7.406	5.920	5.952
6	9.130	8.921	37.330	37.349	6.490	6.533	5.680	5.705
7	7.130	7.143	37.800	37.798	8.090	8.061	6.020	6.002
8	8.160	8.266	37.770	37.791	7.950	7.858	6.100	5.952

St.l.m – Value obtained by standard laboratory method

NIR – Value obtained by NIR method of analysis

During the process of development of calibration model and its validation, certain number of samples had to be excluded in order to obtain the most reliable model possible. Therefore, the number of samples presented in Table 2 is lower than initial number of samples of soybean cake (9 samples for crude fibre and 14 samples for other parameters). This is also reason why, in case of moisture, crude proteins and crude fibre, a model was obtained which gives more reliable results, but with narrower range of contents of determined nutritional parameters. In case of crude fats, however, a calibration model was obtained which gives very reliable results for wide range of concentrations of this constituent. Based on obtained results it can be concluded that even with small number of samples which were tested by using standard laboratory methods of analysis, it is possible, using NIR method, to receive very reliable results, but adequate selection of samples for development of calibration model is of crucial importance. When the number of samples with various concentrations of relevant constituents is higher, a calibration model for wider range of concentrations can be developed, which would also give reliable results.

There are also other ways of implementation of NIR technology, such as use of calibrations offered/provided by manufacturers of NIR devices. However, this way is associated with considerable costs. Also, calibration curves provided by manufacturers of NIR devices, although developed on large number of samples, often need to be adjusted due to high variations in the quality of raw materials.

Conclusion

Introduction of NIR technology is a good strategy of development of facilities for production of animal food, if the complexity of their production process is taken into consideration. NIRS method, as fast analytical method, enables timely receiving of required results, improvement in the quality control and increase in the efficiency of the animal food production, with final outcome in increase of the profitability and competitiveness.

Results obtained by applying NIR method in analysis of the soybean cake samples showed no significant differences compared to results obtained by standard laboratory methods, which proves that NIR method can be very reliable in determination of the composition of raw materials used in production of animal food, as well as of finished mixtures. Also, duration of NIR method of analysis was incomparably shorter than duration of standard laboratory methods.

Primena NIR tehnologije u industriji hrane za životinje

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Rezime

Sve oštrija konkurencija u domenu proizvodnje hrane za životinje zahteva da proces proizvodnje bude što brži i efikasniji. NIRS metoda, kao izuzetno brza analitička metoda, predstavlja dobru soluciju koja poseduje veliki potencijal za unapređenje nadzora i kontrole proizvodnog procesa u fabrikama hrane za životinje. Uzorci sojine pogače analizirani su standardnim hemijskim metodama (referentne metode) i NIR metodom, u cilju poređenja rezultata ove dve metode. Analiziran je sadržaj vlage, sirovih proteina, sirove masti i sirove celuloze. U postupku kalibracije NIR uređaja za ove parametre kvaliteta sojine pogače, ostvareni su veoma dobri koeficijenti determinacije (R^2), što znači da su dobijeni pouzdani kalibracioni modeli. Rezultati dobijeni primenom NIR metode za analizu uzoraka sojine pogače (u postupku validacije modela), nisu se bitnije razlikovali od rezultata standardnih laboratorijskih metoda, što dokazuje da NIR metoda može biti vrlo pouzdana u određivanju sastava, kako sirovina koje se koriste u proizvodnji hrane za životinje, tako i gotovih smeša. Prednosti uvođenja NIR tehnologije u fabrike hrane za životinje su: brza kontrola sadržaja relevantnih sastojaka u sirovinama i gotovim proizvodima, poboljšanje kvaliteta proizvoda, snižavanje troškova, mogućnost ugradnje NIR uređaja u proizvodni proces (at line analiza) - kontinualna kontrola procesa proizvodnje hrane za životinje.

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