

NEAR INFRARED SPECTROSCOPY – AN ALTERNATIVE TO DETERMINE THE CRUDE FIBER CONTENT OF FORAGES

Monica HĂRMĂNESCU¹, Alexandru MOISUC¹, Iosif GERGEN²

¹ Banat's University of Agricultural Sciences and Veterinary Medicine, Faculty of Agriculture, Timisoara, Calea Aradului nr. 119, RO-300645, Romania, monicaharmanescu@yahoo.com

² Banat's University of Agricultural Sciences and Veterinary Medicine, Faculty of Food Technology, Timisoara, Calea Aradului nr. 119, RO-300645, Romania.

Abstract: *In our days NIR spectroscopy represent a promising alternative to the chemical methods for crude fiber contents of forages. The main objective of this study was to obtain a NIR calibration model for prediction this parameter of forages harvested in June 2009 from hill permanent grassland (Grădinari, Caraș-Severin District). The experimental field was organized in ten experimental trials fertilized organic, mineral, and organo-mineral. The floristic composition of forages from this period was determined gravimetrically. From Poaceae were present Festuca rupicola and Calamagrostis epigejos. Fabaceae family was represented by Trifolium repens and Lathyrus pratensis. From other botanical family: Rosa canina, Filipendula vulgaris, Galium verum and Inula britannica. Like input data for NIR calibration were used the results for this qualitative parameter by chemical method and the reflectance values from 150 NIR spectra for all analysed samples. Partial last square (PLS) regression was used to obtain the "NIR - Total Fiber" model, implemented in Panorama program (version 3, LabCognition, 2009). The statistical parameters ($R^2=0.80$; RMSEC=2.73) and the differences between references and predicted values situated in range 0.03 and 9.24% suggest a medium quality of calibration model, but it is promising to use it to predict the crude fiber contents of forages from grassland in this period of year using higher number of samples for calibration.*

Key words: *forages quality, complex fertilizers, PLS- NIR model, grassland.*

Introduction

Since Romania becomes part of European Union the national authorities must adjust the legislation to those European also regarding the quality and safety of the food. The 150/2004 law from our country transpose partially the CE 178/2002 Rule, which establish the most important recommendations on the qualities of raw matter, from vegetal and animal origin, destined to obtain the food [8]. The security and quality of food with animal's origin must be discussed in direct correlation with the requests for forages quality used like animal's feed.

The forages from permanent grassland represent the cheaper source of feed for

ruminants. Fiber content of these forages, alongside of protein, lipids, minerals and vitamins, represents one of the most important parameter which characterize the forages quality [3].

Ruminants have the capacity to digest and use crude fiber like source of nutrients [2]. For example the cellulose content of forages must be in their ratio between 23-25% [1], representing the key in intestinal transit, in stimulation of satiety sensation [2], like energetic source in animal's metabolism [6].

The chemically determination of this parameter request a high reagents consumption, qualified human resources to perform the operation, a long time to obtain the results [7]. An important

alternative for these chemical models is represented in our days by NIR Spectrometry, a non-destructive method which allows obtaining very fast the final results (appreciatively 3 minutes), without reagents consumption, medium pollution and samples destroying [4, 5].

Experimental

The forages were harvested in June 2009 from hill permanent grassland (Grădinari, Caraș-Severin District). The soil of permanent grassland was Calcic Luvisol and the annual average temperature around 10.4°C.

The experimental field was organized in ten fertilized trials in randomized plots, in multiple stage blocks with five replications. It was used for mineral fertilization: 15:15:15 NPK complex, ammonium nitrate, potassium salt, superphosphat). Like organic fertilizer was chose fermented sheep manure. The fermented sheep manure was applied at each two years, even the mineral fertilizers yearly. The fertilization process was made during the period 2003-2008.

The ten trials were: V1-unfertilized trial, V2-20 t/ha sheep manure, V3-40 t/ha sheep manure, V4-60t/ha sheep manure, V5-20 t/ha sheep manure + 50P₂O₅(Kg/ha), V6-20 t/ha sheep manure + 50P₂O₅ (Kg/ha) + 50 K₂O (Kg/ha), V7-20 t/ha sheep manure + 50 P₂O₅ (Kg/ha) + 50 K₂O (Kg/ha) + 50N(Kg/ha), V8-100 N (Kg/ha) + 50 P₂O₅ (Kg/ha) + 50 K₂O

(Kg/ha), V9-150 N (Kg/ha) + 50P₂O₅(Kg/ha) + 50 K₂O (Kg/ha), V10 - (100+100)N (Kg/ha) + 50 P₂O₅ (Kg/ha) + 50K₂O(Kg/ha).

The floristic composition of forages from the ten trials for this period of year was determined gravimetrically. From *Poaceae* family dominant was *Festuca rupicola* (varied between 16.00 – 52.00%), followed by *Calamagrostis epigejos* (5.00-13.00%). Fabaceae family was represented mainly by *Trifolium repens* (dominant) and *Lathyrus pratensis*. From other botanical family were present *Rosa canina* (7.00-18.00%), *Filipendula vulgaris* (3.00-9.00%), *Galium verum* (3.00-7.00%) and *Inula britannica* (5.00%).

NIR calibration model was obtained by PLS (Partial Last Square) regression, implemented in Panorama software (Variant 3, LabCognition, 2009). Like input data were selected the chemical data for crude fiber content, determined by JAOAC 962.09/1990 [7] method (samples are sequentially refluxed in dilute base followed by dilute acid), and the reflectance values from 150 NIR spectra. The V670 Spectrophotometer by Abble-Jasco was the instrument used to scan the spectra in the range 800-2500 nm, and than was selected with Panorama software three spectral ranges favourable to perform the calibration model for crude fiber determination. These spectral ranges were specific for the overtones of fundamental frequencies of OH bound, characteristic for fiber compounds (Table 1).

Table 1.

Calibration data for the „NIR-CF” model with 3 spectral ranges

No.	Selected spectral ranges	Number of wavelengths
1	[1282.5 .. 1433.5]	303
2	[1542.0 .. 1949.0]	815
3	[2263.0 .. 2356.0]	187
„NIR-CF” - NIR-crude fiber model		

Results and Discussion

For all the grounded dried samples the chemical results and NIR spectra were obtained in triplicate.

Statistical parameters for “NIR-CF” model with the three selected spectral ranges are presented in Table 2:

Table 2.

Statistical parameters for „NIR-CF” model with three selected spectral ranges

R ²	0.80
RMSEC	2.73
SD	4.75

These parameters suggest a medium quality of “NIR-CF” model, but were better than the case when were used the entire spectral domain ($R^2 = 0.7355$, RMSEC = 3.28, SD = 4.39). The quality of NIR calibration model is underlined also in the graphical presentation of prediction for crude fiber by „NIR-CF” model with three

spectral ranges (Figure 1) and also by the differences between the chemical results and those predicted for control samples. The control samples were harvested in the same period of year and grassland and conditioned in the same manner with those used to perform the „NIR-CF” calibration model (Table 3).

Table 3.

The results of crude fiber (%) prediction for the control samples forages (June2009) by „NIR-CF” calibration model with 3 spectral ranges

Control sample's name	Crude fiber (%)		
	Real (chemical method)	Predicted (NIR model)	Differences between Real - Predicted
101a	32.10	34.11	-2.01
101b	43.99	34.75	9.24
101c	18.85	24.24	-5.39
101d	31.06	26.92	4.14
102a	34.57	34.38	0.19
102b	24.89	27.08	-2.19
102c	25.46	23.27	2.19
102d	28.25	26.31	1.94
103a	36.45	36.35	0.10
103b	32.08	32.28	-0.20
103c	21.07	23.42	-2.35
103d	28.22	28.68	-0.46
104a	38.85	34.97	3.88
104b	32.98	30.26	2.72
104c	20.72	23.36	-2.64
104d	30.82	26.35	4.47
105a	35.17	34.70	0.47
105b	20.73	29.55	-8.82
105c	24.33	25.05	-0.72
105d	32.08	30.91	1.17
106a	33.01	33.91	-0.90
106b	22.60	27.64	-5.04
106c	24.74	23.06	1.68
106d	31.02	26.77	4.25

107a	36.41	36.30	0.11
107b	29.78	31.26	-1.48
107c	20.03	24.18	-4.15
107d	31.62	29.87	1.75
108a	36.44	37.69	-1.25
108c	22.42	24.18	-1.76
108d	29.55	30.21	-0.66
109a	33.75	38.03	-4.28
109b	28.22	30.83	-2.61
109c	24.37	24.34	0.03
109d	36.07	35.46	0.61

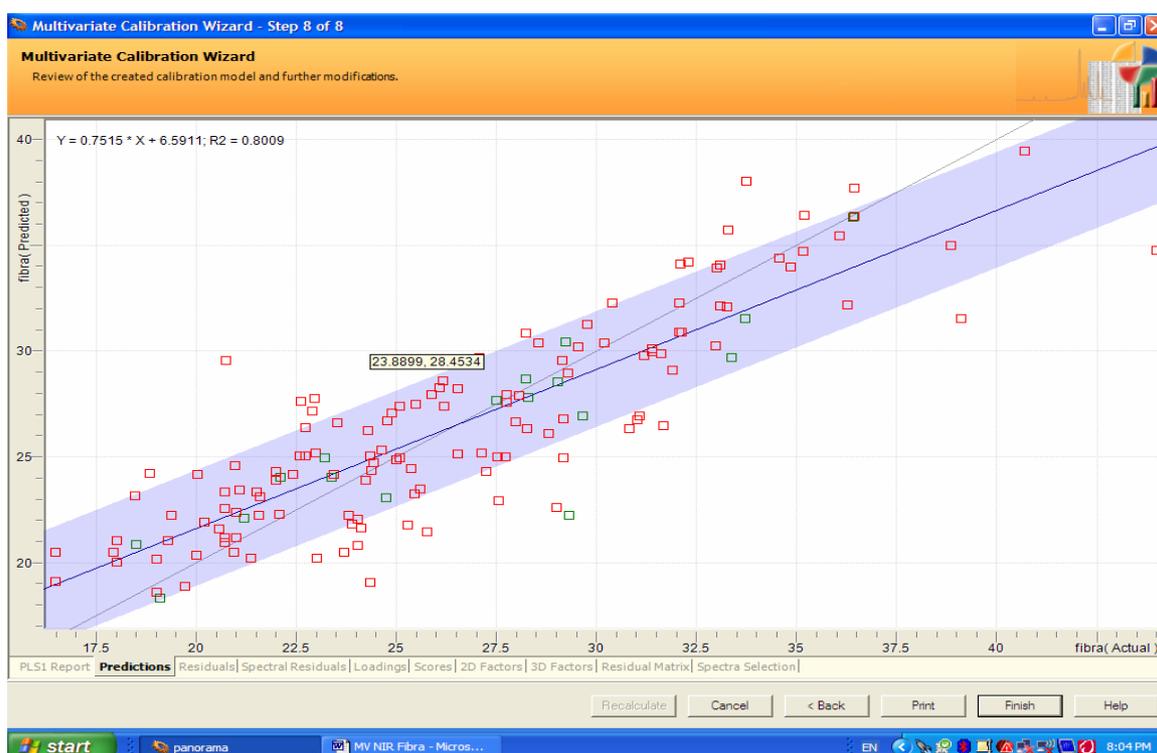


Figure 1. Prediction of crude fiber by the „NIR-CF” model with 3 selected spectral ranges

The differences between values obtained by chemical method and those predicted by „NIR-CF” model were situated between 0.03 and 9.24%. Almost 31.43% from these values were under 1.00%; 40.00% in range 1.01-3.00%; 17.14% between 3.01-5.00%; and 11.43% in range 5.01-9.24%. These results indicate a medium quality of performed calibration model, but encourage us to continue these researches using a high number of samples to characterize better the concentrations of this qualitative parameter of forages from the permanent grassland

Conclusion

The PLS regression model “NIR-CF” with three selected spectral ranges for the determination of crude fiber of forages harvested in June 2009 had a medium quality. But this model promised to be used with success to determine routinely this parameter for the samples harvested in this period of year from the permanent grassland after the enrichment with a higher number of forages samples.

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