TOTAL ASH CONTENT DETERMINATION FROM GRADINARI (CARAS-SEVERIN COUNTY) PERMANENT PASTURE FORAGES USING NIR SPECTROSCOPY

DETERMINAREA CONTINUTULUI TOTAL IN CENUSA DIN FURAJELE PROVENITE DE PE PAJISTEA PERMANENTA DE LA GRADINARI (CARAS-SEVERIN) FOLOSIND SPECTROSCOPIA NIR

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Abstract: *The aim of this scientifically paper was to study the possibility to determine total ash content of forages harvested from permanent pasture from Gradinari (Caras-Severin County) by NIR Spectroscopy. For this it was realized correlations between the values obtained for total ash content using classical dry mineralization method and the reflectance values from NIR spectra. The correlation coefficient R^2 obtained for regression equation when it was used the minimum reflectance values was 0.8077, higher than those for maximum reflectance values, $R^2=0.6356$.*

Rezumat: *Obiectivul acestei lucrari stiintifice a fost studierea posibilitatii de determinare a continutului total in cenusa in probelor de furaje recoltate de pe o pajiste permanenta de la Gradinari (Caras-Severin)) prin spectroscopie NIR. Pentru aceasta s-au realizat corelatii intre valorile obtinute pentru continutul total in cenusa prin metoda calcinarii si valorile reflectantelor din spectrele NIR. Coeficientul de corelatie R^2 pentru ecuatia de regresie in cazul utilizarii valorilor minime ale reflectantelor a fost de 0.8077, mai mare decat cel obtinut in cazul valorilor maxime ale reflectantelor $R^2=0.6356$.*

Key words**:** *total ash content, NIR Spectroscopy, forages*
Cuvinte cheie**:** *continut total de cenusa, spectroscopie NIR, furaje*

INTRODUCTION

For a rational alimentation of human is recommended to consume animal food [MOISUC & DUKIC, 2002]. To obtain a high quality of animal food is necessary to obtain a high quality of feed, with a proteins, carbohydrates, lipids, and mineral contents appropriate with the animal's necessities.

It is known already that minerals have a significant role in the growth and development of plants. For example minerals influence the enzimatic activity, mentain the integrity of cellular system of endo-membranes, are involving in osmotic pressure regulation and in maintenance of acid-base equilibrium, react with the organic compounds and form a high number of complex substances, influence the hydration degree of plasmatic colloids and the reaction of vacuolar solution [SUMALAN, 2006]. The total mineral content accumulate in forages depends by their concentration in soil, water and sometime in air [KABATA-PENDIAS & PENDIAS, 1992].

Chemically, the determination of total minerals content means the determination of total ash content [DRINCEANU, 1994]. In our days the total ash content is determinated using dry mineralization method. To realize this method is necessary to burn a quantity of samples, for few hours, at 500-650^{o}C. Dry mineralization method request time, a high consume of electrical energy, and is limited by the oven surface or the number of quarts capsule that can be burned in

the same time.

NIR Spectroscopy (Near Infrared Reflectance Spectroscopy) is qualitative and quantitative analyze method considered being a very quickly, non-destructive and protective method for environment [WILSON, 1994]. NIR spectroscopy method has an important role in reducing the costs and the time necessary to analyze the samples, and in increasing the number of samples that may be analyzed [GARCÍA & COZZOLINO, 2006]. That's way NIR Spectroscopy can be used like a routine method to predict the total mineral content (total ash content) of forages.

The aim of this study was to determine the total ash content using Near Infrared Reflectance Spectroscopy. For this objective it was harvested forages samples from Gradinari permanent pasture (Caras-Severin County) and was analyzed for total ash content using both NIR Spectroscopy and dry digestion methods. Then it was made correlations between the obtained values for total ash content using dry digestion method and those obtained using NIR Spectroscopy.

MATERIAL AND METHODS

Samples:

Samples were harvested on October 2007, in triplicate, from a permanent pasture from Gradinari, a collinear village situated in Caras-Severin. The main species present on permanent pasture from Gradinari were: *Festuca rupicola* and *Calamagrostis epigeios*. Other species were *Antohoxanthum odoratum, Briza media, Poa pratensis, Trifolium arvense, Trifolium medium, Genista tinctoria, and Lotus corniculatus.* From the other botanical families was presented *Filipendula vulgaris*.

The permanent pasture was organized in ten experimental variants with different doses of fertilization. For each variant were made five repetitions. The fertilization doses for all the ten experimental variants are present in Table 1:

Table 1

The fertilization doses for permanent pasture from Gradinari (Caras-Severin)

Variants	Fertilization doses
GV1	unfertilized
GV2	20 t manure
GV3	40 t manure
GV4	60 t manure
GV5	20 t manure + 50 P
GV6	20 t manure + 50 P + 50 K
GV7	20 t manure + 50 N + 50 P + 50 K
GV8	100 N + 50 P + 50 K
GV9	150 N + 50 P + 50 K
GV10	100 + 100 N + 50 P + 50 K

G – Gradinari; V – experimental variant

Each sample harvested from the ten experimental variants was dried at room temperature (appreciatively 22^{o}C) for two weeks. Then all the samples were grounded. The data for total ash content by dry mineralization method represent the mean of values obtained for harvested samples for each experimental variant.

Determination of Total Ash Content

Total ash content was determined using dry mineralization method. Appreciatively 5 g

of samples were burned in the quartz capsules for 4 hours, at 650^{o} C. For all the samples the determination were made in triplicate.

NIRS spectra were obtained using V 670 Spectrophotometer instrument by Abble-Jasco in the range 800-2500 nm. For all the samples the scan was made in duplicate.

Statistical interpretation of obtained data was performed with Statistica-6 Software.

RESULTS AND DISCUSIONS

The results obtained for total ash content for the analyzed samples, using dry mineralization method, and the maximum reflectance values for main five frequencies from NIR spectra are present in Table 2:

Table 2

Total ash content for the analyzed samples, determinated using dry mineralization method, and maximum reflectance values for five frequencies (L1 – L5) obtained from NIR spectra

Experimental variants	Total ash content %	L1 1325 nm	L2 1650 nm	L3 1853 nm	L4 2017 nm	L5 2175 nm
GV1	10.82	65.3950	61.9823	61.4499	52.1437	48.3262
GV2	10.67	66.2327	63.6254	63.1864	54.2431	49.9864
GV3	8.63	66.9474	65.1285	64.7231	55.3270	51.9850
GV4	8.34	68.1071	65.2311	64.6970	54.5213	50.5113
GV5	9.69	66.2676	63.4140	62.8255	53.4942	49.4864
GV6	8.62	66.7912	63.9073	63.4192	54.4218	50.0608
GV7	10.45	64.3516	61.6213	61.1461	52.2575	48.3815
GV8	8.17	65.9621	63.5955	63.2175	54.4141	50.2327
GV9	8.20	67.8971	65.3148	64.8446	55.3284	51.0317
GV10	7.97	67.4657	64.2720	63.7065	54.1027	49.7582

G – Gradinari; V – experimental variant

The results obtained for samples in this case were interpreted with Multiple Regression Analysis. Using the statistical parameter was established the regression equation for total ash content (%) calculations with maximum reflectance values of the five frequencies is:

$$\textbf{Total Ash Content (\%)} = 53.23 + 0.02L1 + 0.76L2 - 1.54L3 - 0.64L4 + 0.76L5 \quad (R^2=0.6356,\ p<0.3845) \quad [1]$$

Because L1 frequency has a small coefficient it is possible to eliminate it, to simplify the obtained regression equation for total ash content (%) with maximum reflectance values, without modify the R^2 coefficient. The new regression equation for total ash content prediction using four frequencies (L2, L3, L4, L5) is:

$$\textbf{Total Ash Content (\%)} = 53.41 + 0.79L2 - 1.55L3 - 0.63L4 + 0.75L5 \quad (R^2=0.6356,\ p<0.2076) \quad [2]$$

The graphical representation of correlation between total ash content (%) determinated using dry mineralization method and predicted values by regression equation on the basis of four frequencies with maximum reflectance is presented in Figure 1:

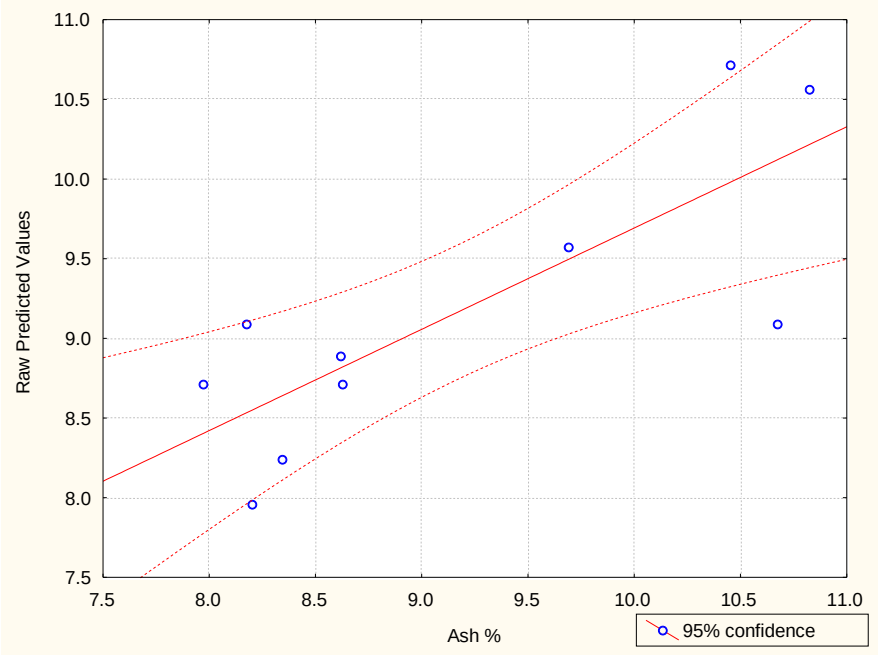

Figure 1. Correlation between total ash content (%) by dry mineralization method and predicted values by regression equation on the basis of four frequencies with maximum reflectance

The results obtained for total ash content, using dry mineralization method, and the minimum reflectance values for main four frequencies from NIR spectra are present in Table 3. The main frequencies with minimum reflectance values were: L1 - 1467 nm, L2 - 1730 nm, L3 - 1930 nm and L4 - 2107 nm.

Table 3

Total ash content for analyzed samples, determinated using dry mineralization method, and minimum reflectance values for four frequencies (L1 – L4) obtained from NIR spectra

Experimental variants	Total ash content %	L1 1467 nm	L2 1730 nm	L3 1930 nm	L4 2107 nm
GV1	10.82	56.2073	59.7144	44.2542	46.8326
GV2	10.67	58.1737	61.2698	47.0139	48.8586
GV3	8.63	59.8141	62.9778	48.0224	51.2464
GV4	8.34	59.3857	62.6463	46.8284	49.9022
GV5	9.69	57.6536	60.9776	45.8836	48.2380
GV6	8.62	58.2396	61.3060	47.1005	48.7289
GV7	10.45	55.9085	59.5121	44.5554	46.9812
GV8	8.17	57.6484	61.3440	46.8536	48.6453
GV	8.20	59.4977	62.7440	47.8669	49.9799
GV10	7.97	57.6659	61.6967	45.8959	48.1168

G – Gradinari; V – experimental variant

The obtained data were interpreted also with Multiple Regression Analysis, using Statistica-6 software. On the basis of statistical parameter, the regression equation for total ash content (%) calculations with minimum reflectance values of the four frequencies is:

Total Ash Content (%) = 71.12 + 1.70L1 - 2.65L2 - 0.51L3 + 0.54L4

(R^2=0.8077, p<0.04893) [3]

The graphical representation of correlation between the values obtained for total ash content (%) by dry mineralization method and predicted values by regression equation on the basis of frequencies with minimum reflectance is presented in Figure 2:

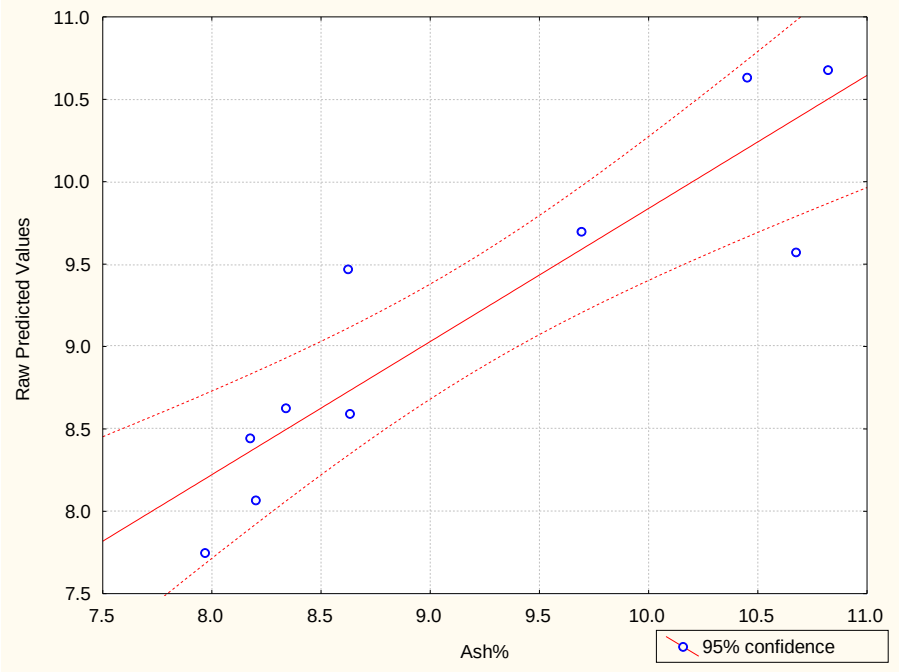

Figure 2. Correlation between total ash content % determinated by dry mineralization method and predicted values by regression equation on the basis of frequencies with minimum reflectance

On the basis of regression coefficients R^2 it is possible to observe that equation [3], obtained for correlation between total ash content values determinated with dry mineralization method and minimum reflectance values from NIR spectra, is better than equation [2], obtained for correlation between total ash content values determinated with dry mineralization method and maximum reflectance, in prediction of total ash content for analyzed forages.

The obtained regression equations for total ash content calculation from NIRS data spectra are true only for the ash values in the range 7 - 11 %. For higher or smaller values it is necessary to obtain a new equation with adequate samples.

These good results, in concordance with those obtained by GARCÍA & COZZOLINO (2006) in South America (Uruguay), motive our future researches in application of NIRS method in total ash content determination using a higher number of forages samples harvested from different pedo – geographically conditions, characteristic for Romania.

CONCLUSIONS

For the analyzed forage samples harvested from Gradinari permanent pasture the best correlation coefficient for total ash content was obtained for the correlation between values obtained using dry mineralization method and minimum reflectance values from NIR spectra, $R^2 = 0.8077$. For the extension of this method in practical application is necessary to investigate a higher number of samples and to validate the method for different pedo – geographically conditions, characteristic for Romania.

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