MOISTURE DETERMINATION FROM COB AND STRAINS USING FT-NIR SPECTROMETRY

DETERMINAREA UMIDITATII DIN STIULETI ŞI TULPINI DE PORUMB FOLOSIND SPECTROMETRIA FT-NIR

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Abstract: *The aim of this paper is to highlight a way to direct analysis method of moisture for maize silo using near infrared spectroscopy in conjunction with multivariable calibration technique. This technique offers a fast and reliable alternative to traditional quantitative method which often takes many hours to complete.*

Rezumat: *Scopul acestui articol este de a evidenţia în mod direct analiza umidităţii porumbului pentru siloz folosind spectrofotoscopia în infraroşu apropiat în conjuncţie cu calibrarea tehnicilor multivariabile. Acestă tehnică oferă o alternativă rapidă şi de nădejde pentru metodele cantitative tradiţionale care de obicei iau multe ore pentru a fi complete.*

Keywords: *NIR, moisture, non-destructive methods, feed, maize*
Cuvinte cheie: *NIR, umiditate, metode nedistructive, furaje, porumb.*

INTRODUCTION

Infrared spectroscopic techniques in combination with chemometrics enable the analysis of raw materials without time-consuming sample preparation methods. Fourier transform infrared spectroscopy has been shown to be a promising tool for the analysis of specific sugars, casein and urea.

NIR spectroscopy has been applied in the food industry and agriculture for determination of water, protein, oil, fat, and carbohydrate contens.

Principal component analysis (PCA) involves a mathematical procedure that transforms a number of possibly correlated variables into a smaller number of uncorrelated variables called principal components. The first principal component accounts for as much of the variability in the data as possible, and each succeeding component accounts for as much of the remaining variability as possible.

MATERIAL AND METHOD

The calibration procedure is based on either a modified form of principal components regression (PCR) or on a partial least squares (PLS) fit for one or more properties. The regression model for each property is refined by selecting only those factors considered to be of statistical significance in determining that property. In PCR and PLS, the spectra are modelled by one set of factors and each property is modelled by relating the concentration values to those factors. In PLS, the spectra are modelled by a different set of factors for each property and the concentration values are modelled by the respective factors.The decomposition step makes it possible to express the data (spectra) as a linear combination of independent terms:

$$\mathbf{X} = \mathbf{S} \times \mathbf{F}^T$$

$$(n_s \times n_v)\ (n_s \times n_f)\ (n_f \times n_v)$$

where:

X is the matrix of calibration spectra (spectra are columns).
F is the matrix of factors (principal components or latent variables), where *T* denotes transpose.
S is the matrix of scores.
n_s is the number of spectra.
n_v is the number of data points (variables) per spectrum.
n_f is the number of factors (principal components or latent variables).

In descriptions of the PLS algorithm, **F** is often referred to as **P**, and **S** referred to as **T**, that is $\mathbf{X}=\mathbf{TP}^T$. If **T** is orthogonal (that is, the scores are independent of each other), then in general the **P** matrix of factors or loadings is not orthogonal, whereas in the PCR case the scores and loadings are orthogonal.

RESULTS AND DISCUSSIONS

All spectra were recorded on a PerkinElmer FT-NIR Spectrometer Spectrum 100N fitted with a „plug-and-play" sampling system accessory for reflectance measurement (NIRA). Eight replicate measurements of each of the calibration samples were collected, and the mean spectrum used for the generation of the calibration equations. Data was collected over the range 10000 to 4000 cm^{-1} at 8 cm^{-1} resolution with 2 cm^{-1} step.

In the same time the moisture content are determinate by classical way using an electrical oven fixed al 105C for 4 hours.

To prove those 4 hours is enough to long to perform moisture calculation we collect a thermo gravimetric curve using a Diamond TG/DTA analyzer. Temperature program is very simple: in first step the sample is heating with constant rate (10^0 C/min) between room temperature and 105^0 C and second step is isothermal step. Fig 1 show the mass-loss as time function and is clear that after 180 minutes the sample mass is stable and all water is loss.

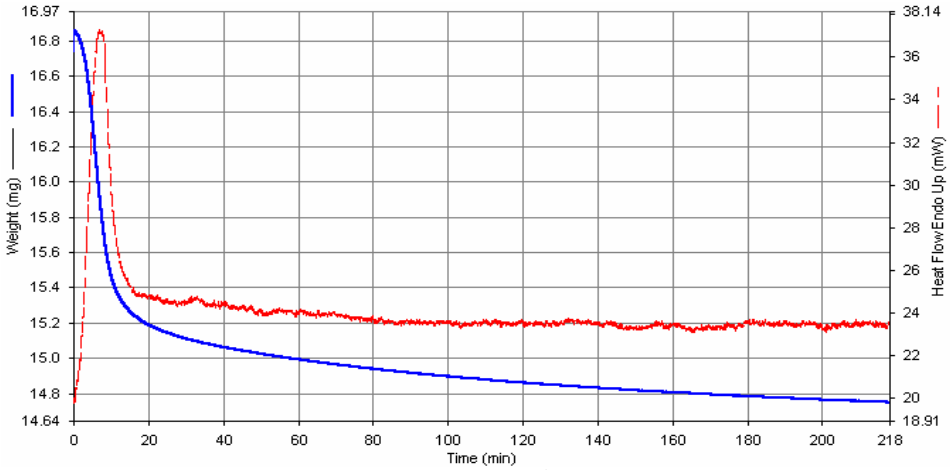

Figure 1 – TG/DTA curve for cob maize

Forty five different samples of maize cob and maize stalks were measured using both electrical oven and NIR spectrometer.. Spectra were recorded by filling a standard sample cup with the sample and scanning in interleaved mode. This mode of operation alternately takes a background spectrum as well as the rationed spectrum which minimizes changes in atmospheric effects

Using these values for spectra we build a mathematical model for direct determination of moisture. For this calculation Spectrum Quant+ v4.60 is used.

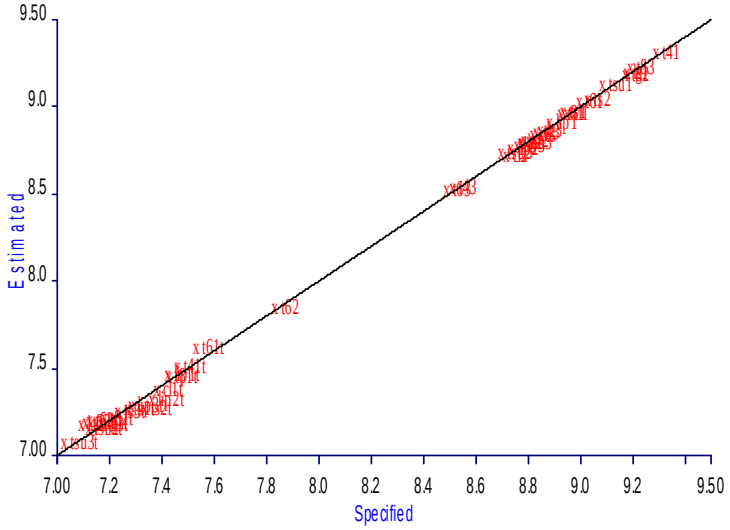

Figure 2- Estimated versus Specified values

A partial least squares analysis (PLS) was performed on the data (45 spectra). It is possible to predict values for moisture in the independent validation set.

Various mathematical pretreatments were tested and a second derivative function chosen to calibration curve and full cross validation is used. Figure 2 show the estimated values versus specified values. Figure 3 show the residual values versus specified values and we see that maximum residual is very low (below 0.1%) .

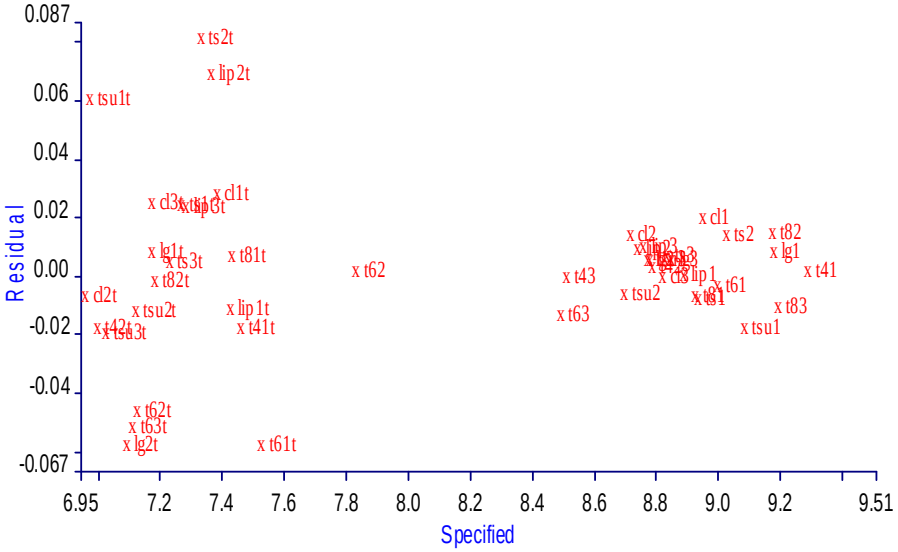

Figure 3 – Residual versus Specified values

The regression model summaries for this method are shown in table 1

Table 1

Summaries of the method

Calculation Parameters:	
Algorithm:	PLS2
Range:	10000 to 4000 cm-1
Interval:	2 cm-1
Analysis Type:	Absorbance
Scaling (Spectra):	Mean
Scaling (Property):	Mean
Smooth:	None
Baseline correction:	Derivative
Order:	2
Width:	5
Normalization:	None
Ordinate threshold:	
Upper threshold:	1.5 A
Lower threshold:	None
Number of factors:	
Minimum:	1
Maximum:	15
Blank regions:	None

CONCLUSION

FT-NIR techniques in combination with chemometrics enable the analysis of moisture in cob and stalk of maize without time consuming. The developed method provides a fast, simple and easily procedure to quantitative determination with good accuracy and precision.

This proves that FT-NIR spectroscopy is an extremely reliable, non-destructive and rapid technique for the quantity of many chemical and physical properties.

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