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## Crude Protein content determination of potatoes by NIRS technology

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### Abstract

The determination of the composition of potatoes is essential in order to meet the current demand for quality products. The traditional methods to measure potato components are both expensive and laborious; therefore, some authors have studied the application of faster and cheaper techniques such as near infrared spectroscopy (NIRS) to determine those components. The objective of the present work is to predict the crude protein (CP) content of lyophilized samples of potato by NIRS. 135 samples were used in this study. NIR spectral data were collected using an AOTF-NIR Analyser (Brimrose) in the reflectance mode. Each sample was scanned twice in the 1100- 2300 nm spectral range. Partial least squares (PLS) regression was applied to the spectral data through Unscramble software (version 8.0.5) to develop a calibration model capable of estimating the CP content of the samples. As a result, correlation coefficients of 0.95, 0.86 and 0.88 were obtained for calibration, cross validation and external validation respectively. Moreover, low standard errors were achieved. The standard error of calibration (SEC) was 0.52, the standard error of cross validation (SECV) was 0.88 and finally, the standard error of prediction was 0.75.

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## 1. Introduction

Potato (*Solanum tuberosum* L.) is considered one of the main food products worldwide being the fourth largest food crop after rice, wheat and maize [1]. Moreover, it represents the staple crop in many of the developing countries around the world nowadays [2]. These tubers are rich in protein, calcium, potassium, and vitamin C, and have an especially good amino acid balance.

Despite that potato products are highly consumed globally; potato industry faces the current demand of quality products by the consumers [3]. The traditional techniques to determine quality and composition of potatoes are chemical methods that imply a considerable amount of handwork and time besides their destructive nature. Therefore, food industry is relying on the adoption of non-destructive and environmentally friendly techniques to determine quality of products [4].

Near-infrared spectroscopy (NIRS) is considered one of the most advanced technologies regarding nondestructive quality assessment techniques [5]. Since its first application in the sixties NIRS technique has been successfully used for the quantitative and qualitative analysis of many agricultural and food products [6]. Moreover, NIRS is considered a green technology due to its nondestructive nature along with the fact that it does not generate any emissions or waste [7]. In the last years, people have become more concerned about protecting the environment driving chemists to look for alternative sample preparation techniques that could reduce the adverse environmental impact of organic solvents. Since NIR is a methodology based on direct measurement of samples avoiding their pretreatment, it is able to reduce the use of solvents and reagent and also the time of analyses [8].

NIRS applications in potato cover both quantitative analyses for the prediction of the different components of the tubers and qualitative analyses to classify samples according to their origin, variety, etc. Since protein content of potatoes is rather small (0.5-2%) comparing to other constituents such as starch and dry matter, it seems difficult its prediction by NIRS. Some authors have investigated the capability of NIRS to estimate protein content in whole potatoes; however, the literature concerning lyophilized samples is scarce.

## 2. Objective

The objective of this study is to investigate the ability of NIRS to nondestructively predict the crude protein (CP) content of lyophilized potato samples.

## 3. Materials and methods

135 lyophilized samples were used in this study corresponding to 135 different varieties of the 2011 season. Chemical analyses to determine CP content and the lyophilization of the samples were carried out at the Basque Institute for Agricultural Research and Development (NEIKER Tecnalia).

For the lyophilization of the samples, tubers were cut lengthwise in order to obtain representative samples of the different tissues. Pieces from 5 to 8 different tubers were then lyophilized in a freeze-dryer Alpha d1-4 (CHRIST, Germany) until they reached 250gr of fresh weight. This process was carried out at -50 °C and 0 atmospheres until the samples lost their whole water content. After that, they were ground with liquid nitrogen up to fine dust and stored at -20 °C until their use.

For the PB determination, first the estimation of total nitrogen content of the samples by Kjeldahl method was carried out. The Kjeldahl method is the standard method of nitrogen determination. It consists of three basic steps: 1) digestion of the sample in sulphuric acid with a catalyst, which results in conversion of nitrogen to ammonia; 2) distillation of the ammonia into a trapping solution; and 3) quantification of the ammonia by titration with a standard solution. Once the percentage of the total nitrogen was calculated, the CP content was determined by the following equation:

$$\text{Crude protein (\%)} = \text{Nitrogen content (\%)} * 6.25 \quad (1)$$

The range of the samples studied covers both white-skinned, red-skinned and blue-skinned varieties. NIR spectral

data were collected using a Luminar 5030 "Hand held" AOTF-NIR (Acousto-Optic Tunable Filter-Near Infrared) Analyzer (Brimrose) in the reflectance mode. A spectral range of 1100-2300 nm with 601 points (2 nm steps) was used to obtain the spectra at room temperature. Samples were scanned twice and the average spectrum was used for the analysis. Each spectrum was an average of 50 scans.

Samples were split in two groups randomly, 90 samples for calibration and 45 for validation. Statistics for CP content of the samples selected for the calibration and validation sample sets are shown in Table 1. The Unscrambler software (ver. 8.0.5 Camo ASA, Norway) was used for partial least squares (PLS) regression.

In order to accelerate the process, no pre-treatment of the data was carried out. PLS regression was performed based on CP content and original spectra (1100-2300nm) of the calibration and validation samples.

A full cross-validation was performed over the calibration set. In this procedure, one sample is removed from the set and a calibration model is developed for the remaining subset. Then, the removed sample is used to calculate the prediction residual. This process is repeated with each sample from the set until all of them have been left out once and finally, the variance of all prediction residuals is estimated [9]

Also, a Principal Components Analysis (PCA) of the data was performed. This is a mathematical procedure that uses an orthogonal transformation to convert a set of observations of possibly correlated variables into a set of values of linearly uncorrelated variables called principal components. The number of principal components is usually less and it could be equal to the number of original variables.

Table 1. Range, mean and standard deviation (SD) of protein content of potatoes.

	Calibration set				Validation set			
	n	Range (%)	Mean (%)	SD (%)	n	Range (%)	Mean (%)	SD (%)
CP	90	6.58-14.12	10.15	±1.67	45	7-14.58	10.01	±1.63

#### 4. Results

The analysis of the data was performed using the PLS regression statistical method. This statistical technique finds a linear regression model by projecting the predicted variables and the observable variables to a new space. In other words, it relates two data matrices, X and Y, by a linear multivariate model. In any case the first step we followed was to analyse the spectra recorded.

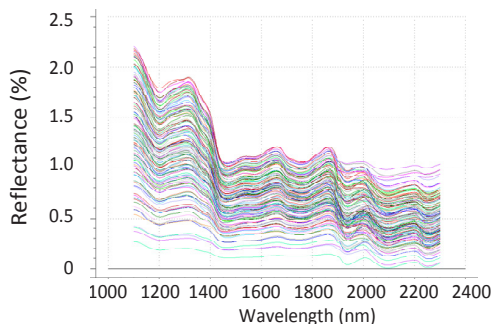


Fig. 1. Reflectance data of the lyophilized potato samples

Figure 1 shows the reflectance spectral data of the 135 lyophilized potato samples.

As it is shown in the Figure 1, all the spectra followed the same trend, showing a sharp peak between 1900 and 2000 nm. According to Law and Tkachuk [10] proteins have three prominent bands in the NH combination region at 1980, 2050 and 2180 nm. Also a noticeable curvature is observed around 1400 nm probably related to starch structures, the main constituent of potatoes [11].

Figure 2 shows the PCA obtained with the raw data. The first principal component (PC1) explains a 96% of the variance while the second (PC2) about 2% which means that almost no information is lost with this transformation. Blue points represent the calibration set of samples whereas red ones correspond to the validation set. As it can be seen in this figure the points are overlapped showing that both the samples in the calibration set and in the validation set are very similar confirming that the division of the groups has been made randomly.

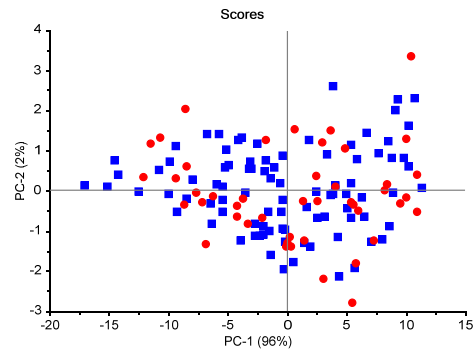


Fig. 2. PCA data of the lyophilized potato samples

Fig 3(a) shows the results obtained with the calibration and cross validation models. The coefficient of correlation ( $r$ ) obtained between spectral data and CP content of the samples was 0.95 and 0.86 for the calibration and cross validation models respectively. The SEC obtained was 0.52 and the SECV 0.88, less than the Standard Deviation of the samples in both cases. The Standard error of laboratory (SEL) was 0.64, lower than the SECV but higher than the SEC.

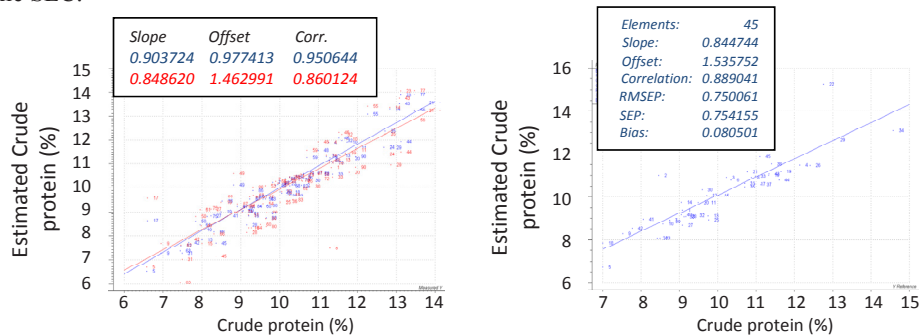


Fig. 3. (a) Predicted CP content versus actual CP in lyophilized potato samples in the calibration data set, (b) Predicted CP content in lyophilized potato samples in the validation data set

Fig 3(b) shows the results obtained with the validation set of samples. An  $r$  value of 0.88 was obtained in this model with a SEP of 0.75.

High correlations for calibration and validation were obtained compared with other authors and the SEC and SEP were relatively small.

Some authors have studied the determination of CP content in mashed and homogenized potatoes by NIRS obtaining relatively low correlation coefficients ranging between 0.36 and 0.78 [12,13]. Authors attributed these low values to the reduced range of this constituent in their samples and the high values of the reference method errors [13].

## 5. Conclusion

Good models have been obtained in this study with high correlation coefficients ranging from 0.86 to 0.95 and low standard errors (between 0.52 and 0.88) both for calibration and validation data sets. Therefore, these results indicate that it is possible to accurately predict the crude protein content of lyophilized potato samples by NIRS. Moreover, NIRS technology is considered a green technique capable of nondestructively predict the quality of many food products.

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