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RESEARCH ARTICLE

Establishing Near Infra Red Spectroscopy (NIR) Calibration for Starch Analysis in Corn Grain

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ABSTRACT

This study has been carried out to calibrate the starch values of 320 corn samples taken from Soil Products Offices (TMO) in seven different geographical regions of by using an NIR instrument. The corn samples used in the study were selected from the regions where corn production is intensive in Turkey and brought to the laboratory. The corn samples brought to the laboratory were milled and then spectra were formed. Subsequently, the starch values were determined in the laboratory with the use of wet chemical analysis methods. The calibration values generated were R= 0.6410; R^2 = 0.4109 Standard Deviation = 4.4208, R = 0.5854 from the validation set; R^2 = 0.3427 Standard Deviation = 4.5662. The calibration interval in the study was 44.53 and 45.72, respectively. This study has concluded that more samples representing the 7 different regions in Turkey for the starch contents of corn are needed in order to generate scientifically reliable results with the FT-NIR device.

Keywords: Corn, Starch, NIR, Calibration

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Mısır Tahılında Nişasta Analizi İçin Near Infra Red Spectroscopy (NIR) Kalibrasyonu Oluşturulması

ÖZ

Bu araştırma Türkiye'nin yedi farklı coğrafi bölgesindeki TMO ofislerinden getirilen toplam 320 adet mısır numunesindeki nişasta değerlerinin NIR cihazı kullanılarak kalibrasyonunu yapmak amacıyla gerçekleştirilmiştir. Araştırmada kullanılan mısır numuneleri Türkiye'de mısır üretiminin yoğun olarak yapıldığı bölgelerden seçilmiştir ve laboratuvara getirilmiştir. Laboratuvara getirilen mısır numunelerinin öğütülmesi işlemi yapılmış, daha sonra spektraları oluşturulmuştur. Akabinde, laboratuarda yaş kimyasal analiz yöntemiyle nişasta değerleri bulunmuştur. Oluşturulan kalibrasyon setinin R=0.6410; R^2= 0.4109 Standart Sapma = 4.4208 şeklinde değerleri alınmış, validasyon setinden ise R=0.5854; R^2= 0.3427 Standart Sapma = 4.5662 değerleri elde edilmiştir. Araştırmada, kalibrasyon aralığı 44.53 bulunurken, validasyon aralığı ise 45.72. olarak bulunmuştur. Sonuç olarak bu araştırma ile Türkiye'nin 7 farklı bölgesini temsil edebilecek mısır örneklerindeki nişasta miktarı FT-NIR cihazı kullanılarak elde edilen kalibrasyonların bilimsel açıdan daha doğru sonuçlar vermesi için daha fazla miktarda numune ile çalışmaların yapılması gerektiğine kanaat getirilmiştir.

Anahtar Kelimeler: Mısır, Nişasta, NIR, Kalibrasyon

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INTRODUCTION

The maize plant is an annual cultivated field crop. It has an advanced root system. Grain maize is a significant role in both human and animal nutrition. Due to its high energy and dry matter, maize has recently been planted as a silage plant as a major feed source for ruminant animals (Anonymous, 2016). Corn is the most produced cereal in the world because of its high energy and nutritional value. Corn production in the world has reached 780 million tons in recent years (Özcan et al. 2009). USA and China are leading countries in the world in corn production (Anonymous 2016). There is a high level of energy in the grain. Corn contains less protein and β-carotene compare to other grains. Corn is used at high levels in poultry and swine rations. In order to raise the energy level of rations, it is used more frequently in the first period of lactation together with grain feed such as wheat and barley. Depending on the animal species and the yield period of the animal, corn grain can be added up to 65-70% of the ration (Anonymous 2016). Near infrared spectroscopy (NIRS) technology is a near infra-red spectroscopy method based on the absorption of electromagnetic radiation in the wavelength range of 400-2500 nm. The nutritional values of the feed raw materials previously calibrated to the device are determined and used to analyze the feed raw materials, and the nutrient content of the feeds is determined quickly. Knowing the values of the feedstuffs involved in the ration directly affects the accuracy of the ration. The tendency towards devices and technologies that make it fast and reliable in determining food values without the need for many chemicals is steadily increasing. Nowadays, near-infrared (NIR) devices have been introduced that give fast results to replace traditional wet chemical analyzes (Cen and He 2007). The NIR device was first used in the world to determine the amount of moisture contained in cereals (Osborne and Feam 1986).

The NIR device is a method of analyzing nutrients such as protein, fat, carbohydrate, ash, moisture (Osborne et al. 1983). NIR technology can also be used to determine the amino acids in industrial feeds (Gonzalez-Martin et al. 2006). Using the NIR device, the results of the analysis were evaluated in corn and the reliability of the results increased after a certain period of time (Jarvis and Walker 1993). NIR technology has also been used to determine the amount of tocopherol in animal feeds (Gonzalez-Martin et al. 2006). Due to the use of external calibrations, nutritional value of feed raw materials produced in Turkey gives misleading results. This situation causes errors in ration calculations (Güngör et al. 2007).

In the NIR device, R^2 value is regarded as a reference value. The R^2 values of 0.95 and above are considered to be fairly good results, whereas those of 0.90 and 0.95 are moderate and those below 0.90 are poor results. For standard error values, the value of 0.3-0.5 is quite good, 1-1.5 is medium, 2-3 is poor (Shenk et al. 2003).

Starch is found in wheat, barley, rye, corn grain in large quantities. Grains are used in abundant quantities so that the amount of energy can be balanced during the feeding of high milk-yielding cows. Generally the optimum starch ratio in dairy cows is 25-30% in dry matter (Dryden 2008). Campbell et al. (1999) estimated the content of starch and cereal amylose in maize with the NIR device. The results show limited sensitivity of this method, whereas the NIR technique can be used as a rough screening method for starch amylose content. In a study (Jiang et al. 2007) to determine protein, starch, and fat content of corn by using NIR device, protein starch and fat contents were found to be the same as those determined with wet chemical method. Same Researchers concluded that the NIR spectroscopy can be used to determine protein, starch and fat content. NIR spectroscopy has been used for the rapid analysis of selected starch samples and the classification of starch molecules is possible with NIR technology (İrudayaraj et al. 2002). In another study, differences between wheat and corn starch were determined using NIR spectroscopy (Hodsagi et al. 2012). There is a certain cost due to the use of time and some chemicals during the chemical analysis. Near infrared reflectance (NIR) can be used to analyze economically in a short period of time without destroying the samples. For example, the NIR technique can be used to analyze complex structures such as starch (Foley et al. 1998). The NIR spectroscopy method has revolutionized the method of analyzing animal feeds quickly and accurately, and the method is becoming more and more popular. Particularly in the field of livestock, when the feed values are determined, the raw materials of feed are vital in predicting nutrients (water, protein, starch, oil, sucrose). In the near future it will be widely used as a feed evaluation method (Givens et al. 1997).

The objective of this study was to establish a national calibration method in Turkey. Another objective was to calibrate the corn starch contents with the use of NIR device.

MATERIAL and METHOD

This study was carried out on 320 grain corn samples. The samples used in the study were collected from Toprak Mahsulleri Ofisi (Soil Products Office) (TMO) in 7 different cities to

represent seven different regions in Turkey. Samples were taken from corn producers with the aid of agencies in seven different provinces such as Adana, Diyarbakir, Gaziantep, Iskenderun, İzmir, Konya and Şanlıurfa. During the sampling process, the identification information of each producer and the information of the region where the corn was grown were recorded. Each corn sample was placed into an impermeable plastic bag after the relevant records were taken and then rapidly transported to Feed Analysis Laboratories. After the samples were delivered to the laboratory, they were milled (ZM200, Retsch Ltd., Düsseldorf, Germany) with ultra-centrifugal rotor mill and passed through a 1 mm sieve. Immediately after this procedure, the ground and eluted corn samples were again placed in clear, impermeable plastic bags and separated. After the grain corn was milled, spectra were taken individually from each sample. During spectral picking, the milled corn samples put on a clear glass petri dish were placed in the automatic rotor of the NIR instrument and the starch-related spectra were collected three times for each corn sample. All collected spectra were stored in electronic form in an appropriate form on the integrated personal computer connected to the NIR device by the same computer program provided by the manufacturer company. The collected samples were again placed into separate plastic bags and stored at -20 ° C until analysis. Milled corn samples were stored in desiccators overnight at room temperature for starch analysis chemometrically and solubilized without rehydration. Starch analysis was carried out with the aid of a digital polarimeter (ADP410 Polarimeter) in accordance with the Starch analysis method (Method No: 996: 11) reported in the AOAC (2018) method for dissolved corn samples. The registration of the wet chemistry results obtained for each sample was carried out separately into the operator program in the NIR Master.

Calibrations and statistical evaluations based on spectral and chemometric analyses (wet chemistry) data obtained with the aid of the NIR instrument in the study were evaluated with the help of the NIRCAL program (Büchi Labortechnik AG, Flawil, Switzerland). Calibration and validation sets within spectra were separated with the help of the program. The obtained spectra were evaluated on the second derivative by PLS (Partial Least Square) method. The SNV (Standard Normal Variate) method was applied to the data for the normalization study. Subsequently, a first grade derivative was obtained (1st Derivation B Cap 5 Points Gap 2) and the data was readied for regression. The outlier values were subtracted from the calibration set and linear regression was applied to the normalized spectra and the calibration quality parameters were manifested. At this stage, the standard deviations of the R^2 value, validation and calibration set were calculated. Graphical outputs were generated with Regression Coefficients according to the reflectance. Furthermore, the squared total value (V-Set PRESS) of the estimation residual error of the validation set was derived. Outlier values were not included in the study because they were determined by the program and reduced the calibration quality.

RESULTS

Reflectance images (1 / log) after preliminary applications applied on the spectra and normalized spectra are shown in Graph 1.

It was observed that the functional spectra obtained after the application were obtained at 9000-4000 nm / cm wave length. The Principal Components of the validation set (V-Set PRESS) has been around 12. The basic component values via the V-Set PRESS are shown in Graph 2.

The resulting regression consistency was obtained as the standard error of the calibration (SEC) divided by the estimation standard error (SEP). The consistency value was determined as between 80 and 100. Consistency analysis results are shown in Graph 3.

The output of the linear regression values obtained from the calibration and validation sets is shown in Graph 4.

Regression analysis included 623 in the calibration set and 205 in the validation set. The model for the Calibration and Validation sets formed as a result of the analysis is indicated below:

Calibration set

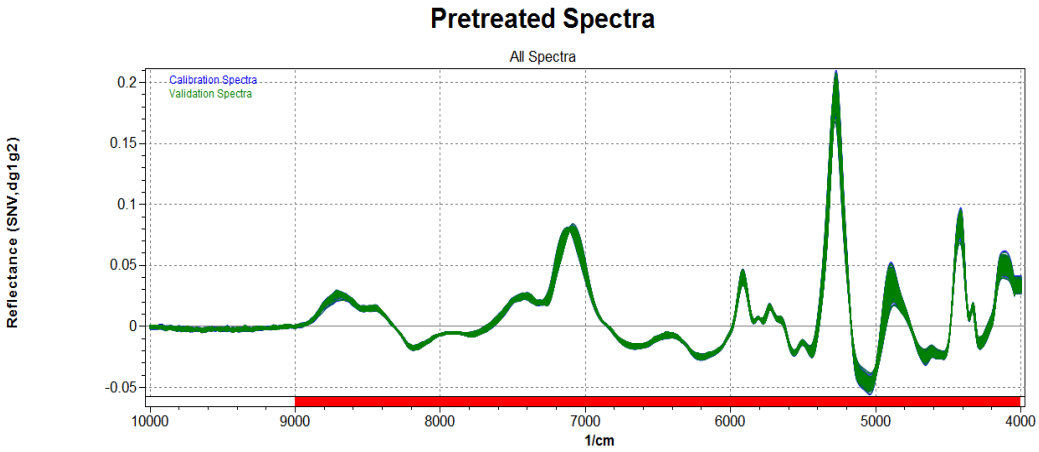
$$f(x) = 0.4109x + 38.4569$$

R=0.6410; R^2= 0.4109 Standard Deviation = 4.4208

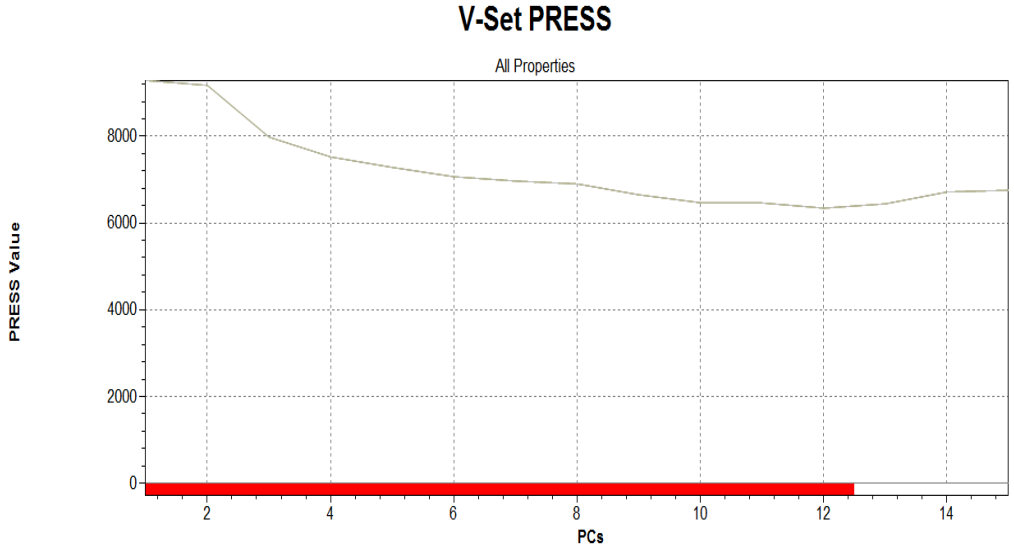
Validation set

$$f(x) = 0.3659x + 41.3845$$

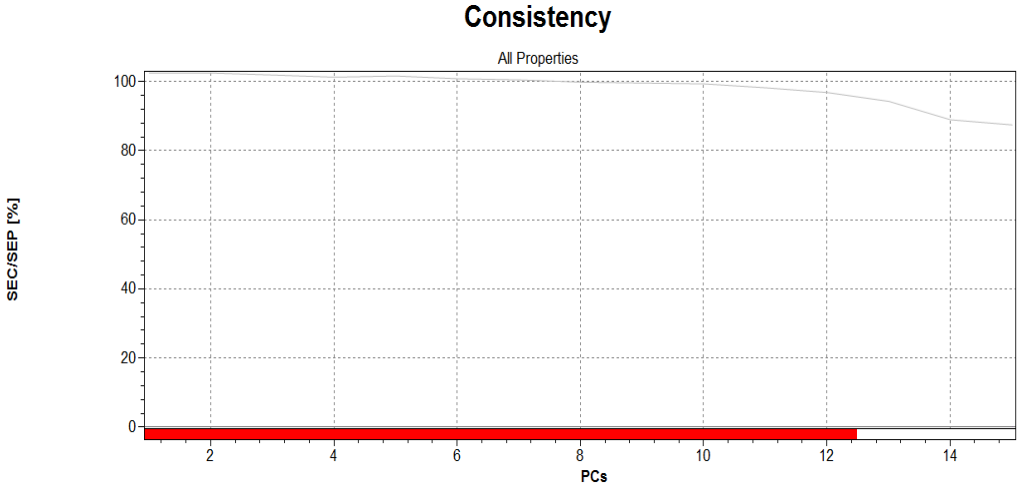
R=0.5854; R^2= 0.3427 Standard Deviation = 4.5662



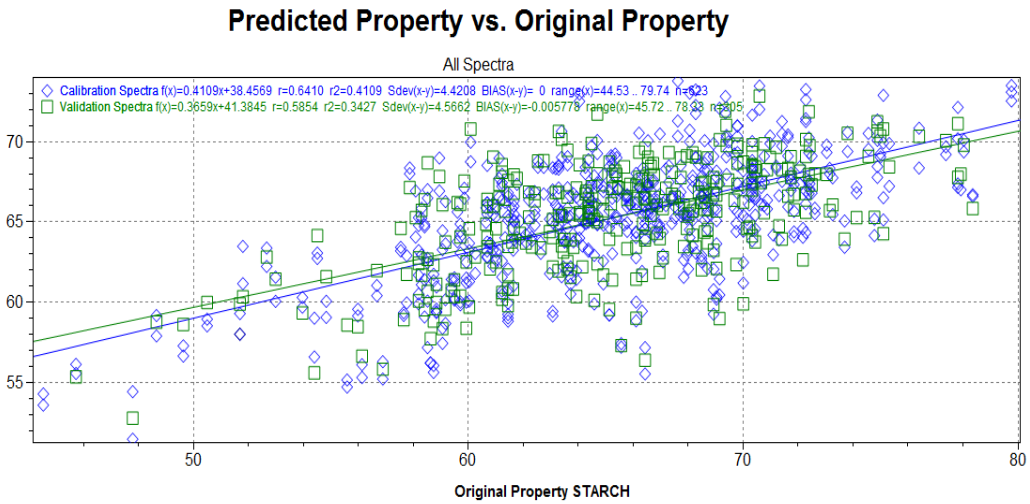
Graphic 1. Normalized spectra set



Graphic 2. Squared total value of the estimation residual error.



Graphic 3. Consistency analysis of calibration set.



Graphic 4. Calibration and validation set models

DISCUSSION and CONCLUSION

A total of 960 spectra from a total of 320 samples (3 spectra from each sample) collected from regions in which corn growing predominates in Turkey have been used in the study. 623 spectra were used in the calibration set and 205 in the validation set. 132 spectra were excluded from the evaluation. The number of spectra and samples obtained in some other studies related to this topic and the values of the data showing the estimation strength and the literature reports are shown in table 1. The results of the chemometric (wet chemical) starch analyzes in the study are shown in Table 2.

As indicated in the table 2, the data set values in this study have been determined with a rather wide interval. This wide range of variation in the values obtained after wet chemical analysis carried out on maize grain samples from different regions of Turkey may be due to starch content values. The starch content of corn in the world also displays quite a comprehensive range interval (Lardy 2013). The grain corn starch content values obtained by wet chemical (chemometric) method in this study were determined as 65.10 %. At the same time these detected values rate among normal starch values for maize in the world and in Turkey (Wehling et al. 1993). Chemometric analyzes were performed on 320 samples in this study and 0.3408 standard error results were obtained. The values we have obtained are the same as the results found in the other study (Wehling et al. 1993). Wehling et al. (1993) determined standard error values for corn starches at an interval of 1.41-2.07% in maize grains. Likewise, Melchinger et al. (1986) reported standard error results with protein, cellulose and water soluble carbohydrates in corn grain, protein and ADF values in corn cobs with NIR analysis which were 0.16%, 0.11%, 0.21%, 0.28% and 0.46% respectively and these values were 6% to 22% higher than the results of chemometric analysis. In a similar study on the subject, the R^2 value in an NIR calibrated study in which corn starch was added to onion salt in different ratios was determined to be in the range of 0.92-0.93 and the standard error calibration value was determined in the range of 2.52-2.65 (Lohumi et al. 2014). Paulsen and Sing (2004) determined that the average extractable corn value in their study of 2267 corn samples carried out between 1997 and 2001 was in the range of 63.4-68.5%. The same authors determined a standard error of 1.24 while the R^2 was determined as 0.79. Baye et al. (2006) reported a corn starch value in the interval of 33.1-75.9% with a standard deviation value of 9.2 and an R^2 value of 0.86. Likewise, Burgers (2009) reported a corn starch value of 60.1% and a standard deviation value of 1.1. The average starch value of sweet potato was found to be 89.79% while the R^2 value was found to be in the range of 0.85-0.92 (Lu et al. 2006), while the mean value of the starch content of corn was reported to be 58.5-74.4% with an R^2 value of 0.77 (Paulsen et al. 2003).

Spectral values were applied as a pretreatment and the first derivate gap2 was applied to the standard normal variation (SNV) and multiplicative scatter correction (MSC) in the study. It is reported that these variables (parameters) are used extensively in the evaluation of spectra (spectrum) (Panero et al. 2013, Zhao et al. 2015). It has been reported that multiplicative scatter correction (MSC) and standard normal variation (SNV) implementations are highly successful in avoiding erroneous path length and light scattering, which can not be determined, by erroneous hardware spectrophotometry errors (Panero et al. 2013).

Variations in ambient temperature variations, sample differences or ambient spectra originating variables due to the sensor may vary (Bokobza 1998). Therefore, in order to avoid this situation formed by the initial impact and minimize the level of the chemometric line variable established by the chemometric analysis and avoid an erroneous evaluation, it is recommended to evaluate the first and second derivatives (Mark and Workman 2003).

The partial least squares method (PLS) was used to evaluate the spectra obtained in this study. This method is widely used in NIRS calibrations and it is reported to be one of the most functional ones (Blanco and Peguero 2012). With this PLS method, explanatory variables with multiple linear correlation between them can be obtained with the help of Harezmi path (algorithms) as well as change in explanatory variables (Via et al. 2014).

The R^2 (coefficient of determination) value is used to calculate the estimation value. This value is the regression coefficient, which is the coefficient of determination of the association between variables and is calculated by taking the square value of R (Sohn et al. 2006). This R^2 value is between 0 and 1. The closer this value is to the number 1, the stronger the result is (Sohn et al. 2006). In the presented this study, the calibration value set for the starch value was 0.6410 and the R^2 value was 0.4109, the R value of the validation set was 0.5854 and the R^2 value was 0.3427. Wehling et al. (1993) found the R^2 value for starch in the range of 0.872-0.892 in their study with maize kernels using the NIRS technique. Likewise, Fu (2004) determined the R^2 value for corn grain starch to be 0.991. In another study (Holder 2012) the aim was to determine the starch amylose and amylopectin ratio with NIRS and the R^2 value was determined as 0.832. The literature reports have significantly higher mean values for corn starch and R^2 values than the R^2 for starch in our study. This conclusion can be attributed to the variety and type of soil in the area where the maize plan was grown, differences in climate and geographical properties and most importantly differences in the races of the maize plants.

This study was carried out with the intention of establishing a national calibration method for maize kernels grown in our country. To this end, maize samples were collected from seven different provinces to represent all regions which were processed with an FT-NIR. It has been concluded that it is possible to use an NIR device to determine the corn starch and set a calibration of a national starch amount to represent the whole country, however in order to have more reliable and usable results a more comprehensive study involving at least 500 and more maize samples must be carried out.

Table 1. Data from a limited number of studies involving R^2 values of nutrients in grain maize.

Corn	Number of Sample	Analyses of Feedstuffs	R^2	Reference
Grain	100	Starch	0.83-0.94	Plumier et al. 2013
Grain	90	Starch	0.98, 0.99	Zhong et al. 2016
Grain	304	Starch	0.77	Paulsen et al. 2003
Grain	90	Starch	0.86	Baye et al. 2006
Grain	2267	Starch	0.79	Paulsen and Singh 2004
Grain	2160	Starch	0.86	Baye et al. 2006
Grain	87	Protein, fat, sugar	0.75, 0.29, 0.02	Tallada et al. 2009
Sorghum, Grain	140	Starch	0.91	Wolfrum et al. 2013
Grain	320	ADF	0.30	Gümüştaş and Bayram 2018

Table 2. Wet chemical starch results in maize rains.

Descriptive statistics of Starch chemistry analysis results	
Average	65,1090
Standard Error	0,3408
Standard Deviation	6,0384
Median	65,4090
Variance	36,4619
Relative standard deviation	0,09274 (9,27%)
Optimum value	79,74
Lowest value	42,58

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