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Araştırma Makalesi

Estimation of Some Quality Parameters of Durum Wheat with Near-Infrared Spectroscopy (NIRS)

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Abstract

The aim of this study was to determine the durum wheat quality parameters using Near-Infrared Spectroscopy (NIRS) at early stages. In this study, Foss NIRSystem XDS was used. Durum wheat types at late stages which were subject to adaptation and plant breeding from test field of East Mediterranean Agricultural Research Institute and some standard varieties were used. In order to determine the reference values needed to construct calibration in NIRS analysis, durum wheat samples were analyzed by classical analysis methods. Moisture, gluten, protein and starch contents, hectoliter weight, and zeleny sedimentation values were therefore determined. Coefficients of determination (RSQ) between classical analysis results and values predicted by NIRS were calculated as follows: for moisture content 0.816, for hectoliter weight 0.906, for gluten content 0.972, for protein content 0.975, for starch content 0.929 and for zeleny sedimentation values 0.968.

As a result, this study shows that NIRS is possibly a fast and efficient method to determine the quality parameters of durum wheat, and can be used for the determination of wheat quality from beginning to the last stage of plant breeding process successfully, in order to supply customer with products in desired quality characteristics.

Keywords: Durum wheat, gluten, NIRS, protein, quality

Makarnalık Buğdayın Bazı Kalite Parametrelerinin Yakın Kızılötesi Spektroskopisi ile Tahmini (NIRS)

Özet

Bu çalışma makarnalık buğday ıslahçılarının ihtiyaç duyduğu makarnalık buğday kalite parametrelerinin erken kademelerde NIRS ile belirlenmesi amacıyla yürütülmüştür. Çalışmada makarnalık buğdayların spektrumlarının alınmasında Foss NIRSystem XDS near-infrared Rapid Content Analyser cihazı kullanılmıştır. Materyal olarak ise Doğu Akdeniz Tarımsal Araştırma Enstitüsü deneme alanında adaptasyon ve ıslah çalışmaları yürütülen ileri kademedeki makarnalık buğday hatları ve bazı standart çeşitler kullanılmıştır. NIRS'da analiz kalibrasyonu üretmek için referans değerleri belirlemek amacıyla makarnalık buğday numunesinin klasik analiz yöntemleri ile kalite parametrelerine ait değerleri belirlenmiştir. Bu amaçla numunelerde nem, gluten, protein, nişasta, hektolitre ve zeleny sedimantasyon analizleri yapılmıştır. Klasik analiz sonuçları ile NIRS'da tahmin edilen değerler arasında elde edilen kalibrasyon belirleme katsayısı (RSQ) nem oranında 0.816, hektolitrede 0.906, glutende 0.972, proteinde 0.975, nişastada 0.929, zeleny sedimantasyonda ise 0.968 olarak hesaplanmıştır. Sonuç olarak bu araştırma, NIRS'ın makarnalık buğdayların kalite parametrelerinin belirlenmesinde hızlı ve etkili bir yöntem olabileceğini, tüketiciye arzu ettiği kalitede ürün sunabilmek için buğday kalite analizlerinin ıslah sürecinin başından son kademesine kadar başarılı bir şekilde kullanılabileceğini göstermektedir.

Anahtar Kelimeler: Makarnalık buğday, gluten, NIRS, protein, kalite

1.Introduction

Wheat is the most widely planted crop in Turkey. Agricultural lands used for wheat constitute 51% of total agricultural lands in Turkey. Although both cultivation area and the amount of wheat produced vary each year, annually 7.5-8 million hectares are used for wheat cultivation and 20-21 million tons of wheat are produced (TSI 2017). Additionally, annually 1.2-1.3 million hectares are used for durum wheat cultivation and 3.5-4 million tons of durum wheat are produced (TSI, 2017). Wheat also plays an important role in the nutrition of Turkish people. It supplies 52% of daily energy need. In our country, a person consumes 205 kg wheat per year in average (80-85% as being bread) (TSI 2017) and consumes 8.5 kg pasta per year in average (TMSD 2017). Durum wheat has received great attention by researchers, producers and consumers. In order to provide consumers with products in desired quality, it is necessary to perform wheat quality determinations from beginning of breeding to the final product.

Quality in wheat is the compliance of wheat for different production purposes rather than having standard characteristics. Cultivation region and climate which changes year to year severely affect the wheat quality. Wheat quality characteristics can be classified as physical, chemical, physicochemical, rheological and technological quality characteristics. The physical quality characteristics include amount of foreign materials, hectoliter weight, vitreousness, grain hardness, grain size, grain color and thousand-kernel weight whereas moisture, ash and protein contents are important chemical characteristics. The most commonly used physicochemical quality characteristics however are falling number, wet and dry gluten contents, gluten index, zeleny sedimentation, Pelshenke value and Berliner-Koopmann test. Rheological characteristics are determined by farinograph and extensograph. Technological quality characteristics include milling yield, bread, semolina, pasta and biscuit quality characteristics. Several methods and instrumentations have been developed in order to evaluate physical, chemical, rheological and baking characteristics of wheat and flour.

Recently there has been increasing interest on quick, reliable and green technologies in both food production and food analyses. Therefore new techniques alternative to traditional

methods have been developed. One of these techniques is NIRS (Cen and He 2007). Quick, reliable and environment-friendly NIR Spectroscopy, which can also be used in food analyses after proper calibration, is based on the measurement of absorption of electromagnetic radiation between 400 and 2500 nm wavelengths (Davies and Granth 1987).

NIRS which was firstly used for the determination of moisture in agricultural products (Norris 1964) is now used to determine pathogens in milk, milk products, meat and egg (Perez-Vich et al. 1998), to evaluate the main quality characteristics such as protein, carbohydrate and oil contents of food and agricultural products and to detect quality characteristics like freshness, color and maturity by analyzing water, ethanol, sugar, organic acids, fatty acids and phenolic contents of food and agricultural products (Sundaram et al. 2010a,b).

Grain yield in durum wheat (Ferrio et al. 2004), durum wheat adulteration detection (Cocchi et al. 2006), amylose and protein content in durum wheat (Lavine et al. 2014), wheat milling characteristics (Blazek and Hruskova 2005), protein and moisture contents (Osborne and Fearn 1983), wet and dry gluten contents (Ertugay et al. 2007), zeleny sedimentation values (Hruskova and Famera 2003), SDS sedimentation values, mixograph peak resistance, gliadin and glutenin contents (Delwiche et al. 1998), color (Dowell et al. 2006), ash content, starch damage (Osborne and Douglas 2006; Miralbes 2004), water absorption, dough strength (Miralbes 2004), properties of fermented dough (Jirsa and Hruskova 2005) and rheological properties of dough (Alava et al. 2001) have been studied using NIR spectroscopy. Additionally NIRS has been applied to determine energy contents of several cereals (Kays and Barton 2002), structural changes during the storage of bread (Xie et al. 2003), wheat types (Carlos 2008) and quality of breads. The data valuable for practical applications have therefore been collected.

Recently NIR spectroscopy has been widely used in every stage in which the determination of wheat quality characteristics is required. NIR spectroscopy is a non-destructive method and quality analyses can be performed without the need for chemicals and qualified personnel. In this environment-friendly method, analysis takes less than a minute

and results for more than one parameter are collected. Although NIR spectroscopy has been used for the determination of quality characteristics of wheat over 50 years, studies on the collection of NIR spectra of durum wheat and correlation of NIRS spectra to wheat quality characteristics are limited in our country. Since present studies are focused on the durum wheat types, the determination of early-stage quality characteristics of high-quality varieties required by breeders during wheat breeding is needed. This study is performed to determine early-stage durum wheat quality characteristics needed by durum wheat trading and processing institutions and establishments, and especially wheat plant breeders.

2. Materials and Methods

Durum wheat types at late stages which were subjected to adaptation and plant breeding from test field of East Mediterranean Agricultural Research Institute and some standard varieties were used in this study. Two hundred durum wheat samples were subjected to classical analyses in order to determine quality characteristics to be used as reference values in NIR spectroscopy calibrations. Moisture, protein and gluten contents were therefore determined according to AACC methods (AACC 2000). Zeleny sedimentation was performed according to ICC standards (ICC 1994). Starch and hectoliter weight analyses however were performed according to Ozkaya and Kahveci (1990).

NIRS system XDS Near-Infrared Rapid Content Analyzer (FOSS NIRSystem, Denmark) and ISI scan program were used to measure spectra of samples which had been analyzed using classical methods. Samples were not subjected any pretreatment. Spectra of durum wheat samples between 400 and 2500 nm wavelengths were scanned by measuring absorbances at every 2 nm intervals.

Calibration equations were constructed using WinISI III v1.61 (Infrasoft International) program. Modified partial least squares method (MPLS) was used to construct calibration equations (Blanco and Villarroya 2002). The best fitting mathematical model was obtained by using several mathematical models in order to correlate the results of the reference analytical methods to the results obtained by NIR spectroscopy. Standard error of prediction (SEP), bias,

slope and correlation coefficient (RSQ) were used to develop calibration model and to determine precision of the validation.

3.Results and Discussion

Calibration and validation statistics performed using the results of reference methods and NIRS for moisture and protein contents, zeleny sedimentation values, starch content and hectoliter weights of durum wheat samples are given in Table 1.

Table 1. Calibration and validation statistics for several quality characteristics of durum wheat samples.

Parameter	Mean±SD	Min (%)	Max (%)	RSQ (R^2)	SEP	Bias	Slope
Moisture (%)	11.98±0.42	11.2	13.0	0.816	0.160	0.000	1.000
Protein (%)	12.11±1.20	9.6	16.9	0.975	0.199	-0.000	0.997
Gluten (%)	26.89±3.57	19.14	39.48	0.972	0.615	0.000	1.000
Zeleny Sedim. (mL)	44.36±9.82	23	82	0.968	1.855	0.000	1.000
Starch (%)	76.64±1.13	71.4	79	0.929	0.320	0.000	1.001
Hectoliter (kg/hL)	81.59±2.31	74.4	85.8	0.906	0.672	-0.013	1.006

SD: Standard deviation, RSQ=R^2 Correlation Coefficient, SEP Standard Error of Prediction

NIR spectra of durum wheat samples are given in Figure 1. Absorption spectra of the samples between 900 and 1400 nm were almost similar. However, at higher wavelengths, spectra of the samples became distinctive.

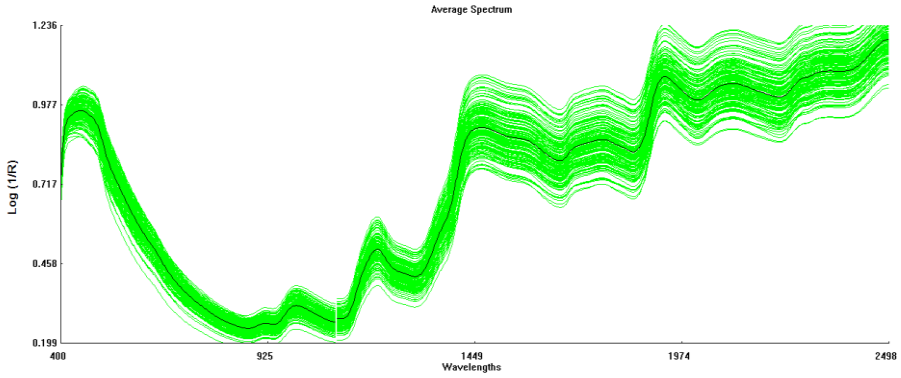

Figure 1. Foss NIRSystem XDS spectra of durum wheat samples.

Correlations between the results measured by reference analytical methods and NIR spectroscopy for moisture, protein and gluten contents, zeleny sedimentation, starch content and hectoliter weight are given in Figures 2, 3, 4, 5, 6 and 7, respectively.. Slope and correlation coefficient for each parameter are given in the respective figure. Higher correlation coefficients resulted in slopes values closer to 1. This shows that predicted values by NIR spectroscopy are closely related to values measured using reference analytical methods.

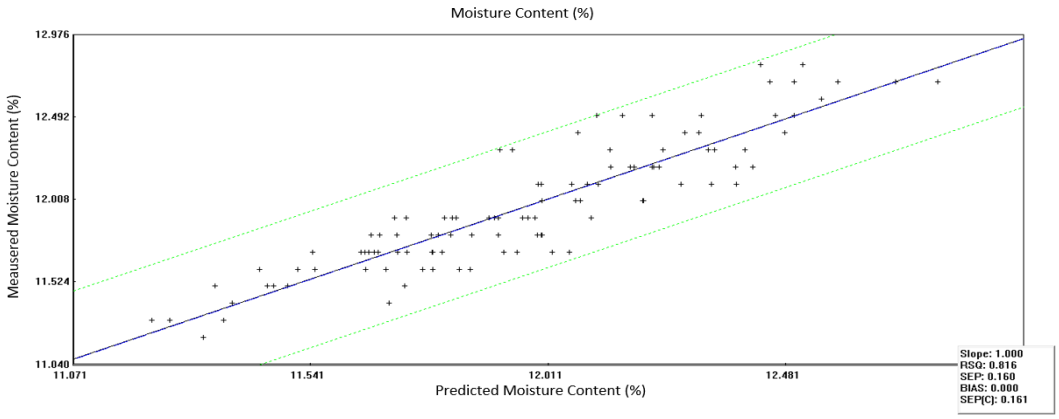

Figure 2. Correlation between moisture contents measured by reference methods and predicted by NIRS

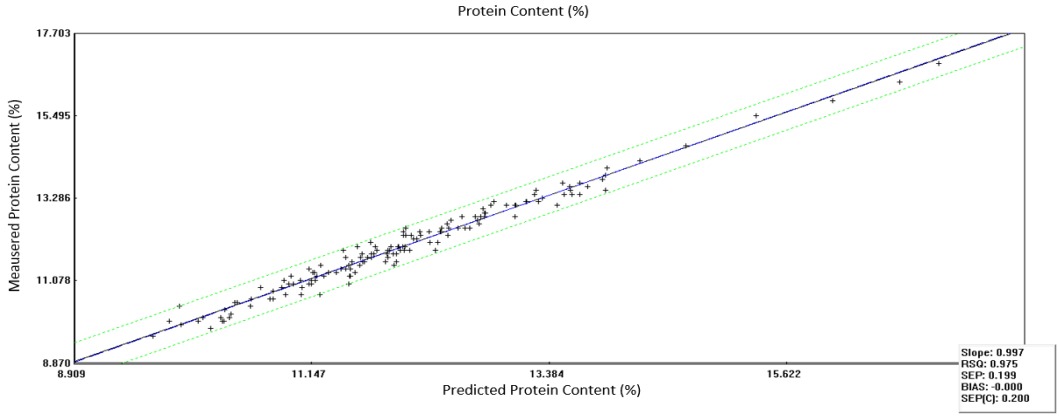

Figure 3. Correlation between protein contents measured by reference methods and predicted by NIRS

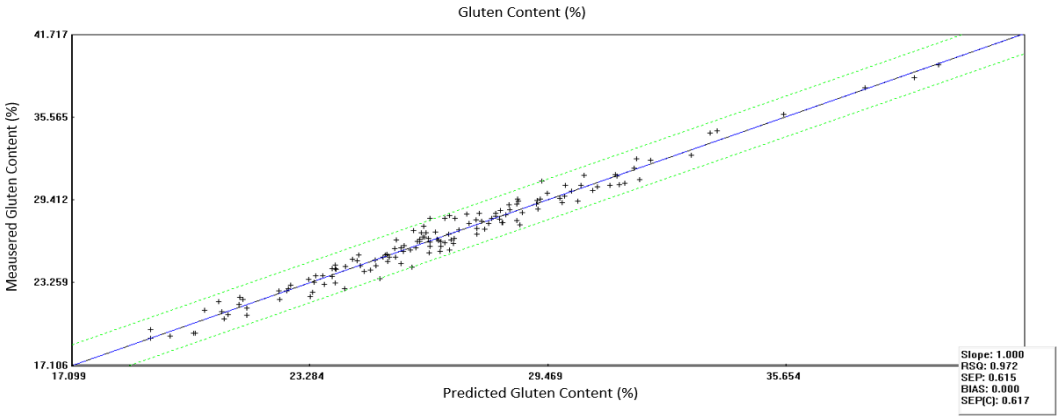

Figure 4. Correlation between gluten contents measured by reference methods and predicted by NIRS

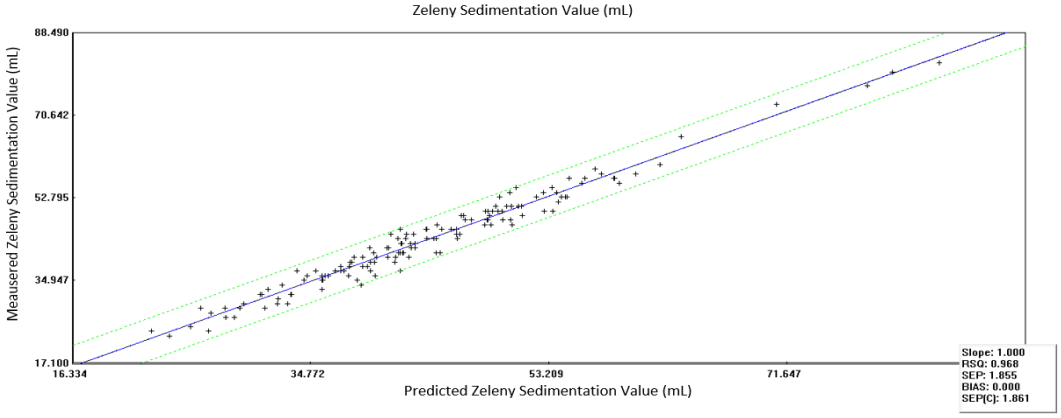

Figure 5. Correlation between zeleny sedimentation values measured by reference methods and predicted by NIRS

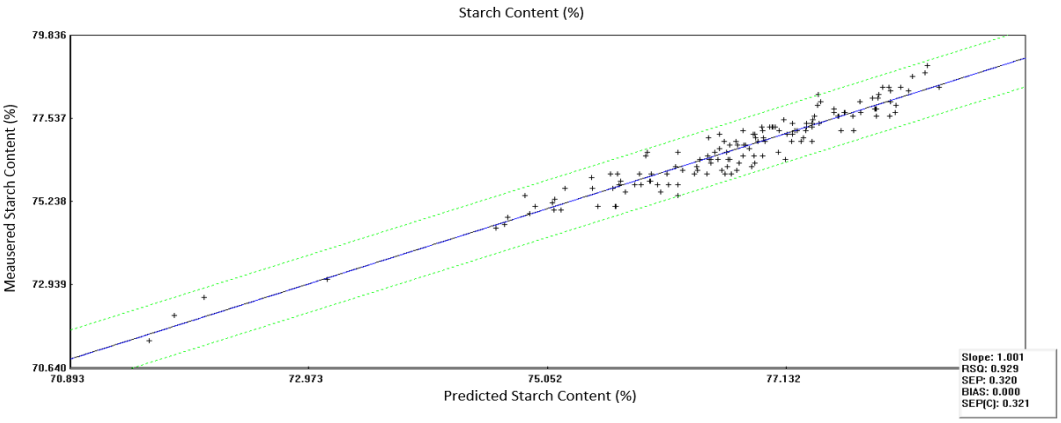

Figure 6. Correlation between starch contents measured by reference methods and predicted by NIRS

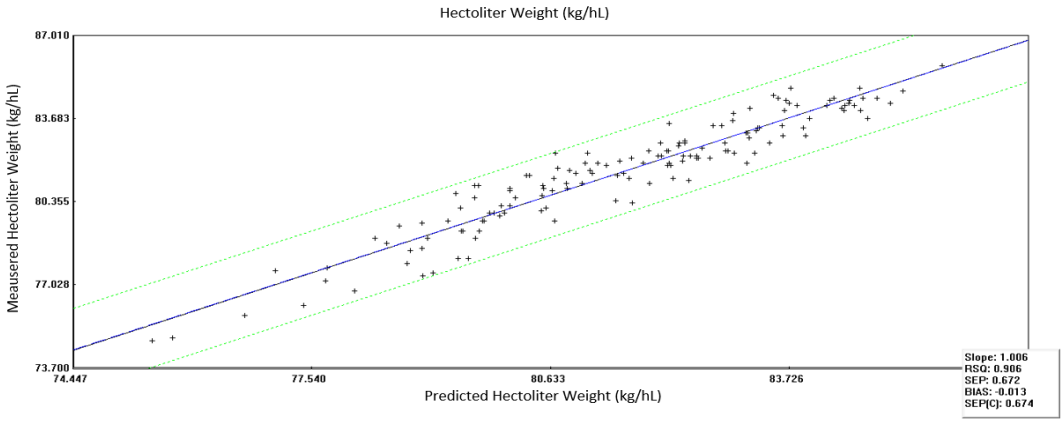

Figure 7. Correlation between hectoliter weight measured by reference methods and predicted by NIRS

As it can be inferred from Table 1, moisture content of durum wheat samples varied between 11.2 and 13%. Average moisture content however was found as 11.98%. Protein contents of the samples were found to range between 9.6 and 16.9% with an average protein content of 12.11%. Gluten contents were between 19.14 and 39.48%. Average gluten content of durum wheat samples was calculated as 26.89%. Zeleny sedimentation value was calculated as 44.36 mL in average (minimum 23 mL and maximum 82 mL). Starch content of durum wheat samples were found in the range of 71.4 and 79% and calculated as 76.64% in average. Hectoliter weights of the samples were in the range of 74.4 kg/hL and 85.8 kg/hL. Average hectoliter weight was found as 81.59 kg/hL (Table 1).

Correlation coefficients between the results of reference analytical methods and values predicted using NIR spectroscopy were found as 0.816, 0.975, 0.972, 0.968, 0.929 and 0.906 for moisture, protein and gluten contents, and zeleny sedimentation values, starch content and hectoliter weight, respectively. Standard errors of prediction for moisture, protein and gluten contents, zeleny sedimentation values, starch content and hectoliter weight were calculated as 0.160, 0.199, 0.615, 1.855, 0.320 and 0.672, respectively. Bias is determined according to the difference between the average of the results obtained by reference analytical methods and NIRS. Bias should be close to zero. When the slope of calibration line is equal to 1, it is at the

equal distance to reference and predicted values. For a certain calibration model, when the standard error of prediction close to zero and correlation coefficient is close to 1, such a model is preferred (Başlar 2008). In this study, bias values were calculated as 0.000, -0.000, 0.000, 0.000, 0.000 and -0.013 for moisture, protein and gluten contents, zeleny sedimentation values, starch content and hectoliter weight of durum wheat samples, respectively. Slopes of the calibration lines for moisture, protein and gluten contents, zeleny sedimentation values, starch content and hectoliter values were however found as 1.000, 0.997, 1.000, 1.000, 1.001 and 1.006, respectively. The results of calibration and validation statistics show that there is a close correlation between the results measured by reference methods and values predicted by NIRS.

Lavine et al. (2014) developed calibration model for the prediction of protein content of waxy condition of durum wheat in their study using NIRS. They found R^2 value as 0.94 for protein content. R^2 value calculated in this study was higher than that reported in the study of Lavine et al. (2014).

Baslar and Ertugay (2011) used NIRS to determine quality characteristics of wheat flour. RSQ values of correlation between the results obtained by reference methods and values predicted using NIRS were found as 0.985 for protein content, 0.976 for wet gluten content, 0.953 for dry gluten content and 0.924 for zeleny sedimentation values. RSQ values calculated in this study were almost similar to the RSQ values stated in Baslar and Ertugay (2011). RSQ value calculated for zeleny sedimentation in this study however were found to be greater than that found by Baslar and Ertugay (2011).

In the study of Jirsa et al. (2008) on the determination of milling and baking properties of bread wheat using NIRS, the highest R^2 values were found for protein and zeleny sedimentation as 0.812 and 0.600, respectively. R^2 values found for protein content and zeleny sedimentation in this study were found to be higher than those reported by Jirsa et al. (2008).

Dowel et al. (2006) reported R^2 values as 0.97, 0.93, 0.57, 0.47, 0.05, 0.87, 0.13 and 0.76 for protein and moisture contents, hardness, ash content, SDS sedimentation values,

gluten content and index and total glutenin content using FOSS 6500 NIRS system in their study performed on the determination of wheat quality characteristics by four different NIRS instruments. R^2 values calculated for protein contents in this study were similar to those reported by Dowel et al. (2006). R^2 values for gluten contents calculated in this study were found to be greater than those reported by Dowel et al. (2006). However R^2 values for moisture contents calculated in this study were found to be lower than those reported by Dowel et al. (2006).

Jirsa et al. (2007) developed calibration models for the prediction of protein content and zeleny sedimentation values of flour in their study on the determination of baking quality characteristics using NIRS. They found R^2 values as 0.991 for protein content and 0.905 for zeleny sedimentation values. R^2 values calculated in this study were in agreement with those reported by Jirsa et al. (2007) yet R^2 value predicted for zeleny sedimentation in this study was higher than that reported in the study of Jirsa et al. (2007).

Miralbes (2004) used near-infrared transmittance spectroscopy (NITS) for the determination of quality characteristics of flour samples in milling industry. R^2 values between the results of reference analyses and NIRS predicted values were found as 0.99, 0.99, 0.96, 0.99 and 0.98 for protein, moisture, dry gluten, wet gluten and ash contents, respectively. In the study performed by Miralbes in 2003, NITS were also used to determine several quality characteristics in wheat. R^2 values in the study of Miralbes (2003) were reported as 0.99, 0.99, 0.95 and 0.96 for protein, moisture, wet and dry gluten contents, respectively. Our results were in good agreement with the results reported in Miralbes (2004) and Miralbes (2003).

Hruskova and Famera (2003) performed a study in order to determine quality characteristics of flour samples prepared from commercial wheat and several wheat types. The highest R^2 values for protein and moisture contents and zeleny sedimentation values were found to be 0.994, 0.983 and 0.749, respectively for the flour samples prepared from several wheat types. For commercial flour samples, the highest R^2 values were reported as 0.968 for moisture content, 0.990 for protein content and 0.749 for zeleny sedimentation values.

Correlation coefficients found in our study were almost similar to those reported by Hruskova and Famera (2003) but R^2 value for zeleny sedimentation calculated in this study were greater than that reported in the study of Hruskova and Famera (2003) whereas R^2 value for moisture content calculated in this study were found to be lower than that reported in their study.

4.Conclusion

The results of this study showed that NIRS can be used as a fast and efficient method requiring small amounts of samples compared to conventional chemical analyses for the determination of quality characteristics of durum wheat planted in our country. Since no chemical is used in NIRS analysis, it is also considered as an environment-friendly method. Calibration and validation results obtained in this study confirm that NIRS can be used to predict quality characteristics of durum wheat. This study therefore suggest that NIRS can be used in all stages of wheat breeding from early to final stages as well as in wheat trading and marketing. Once the proper calibration is obtained, the other properties of durum wheat and the quality characteristics of various food and agricultural products can also be determined by using NIR spectroscopy.

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