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Data-Driven Evaluation of Near-Infrared Spectroscopy in Grain Quality Control

HASAN ASKARLI
Department of Quantitative Methods, University of Pannonia, Egyetem str. 10, Veszprem, 8200, Hungary
haskarli@phd.gtk.uni-pannon.hu

ABSTRACT

This study highlights the quantitative assessments of near-infrared spectroscopy for the analysis of quality parameters of bread wheat. The focus is on analytical performance. Statistical tools, Cochran's test and z-score analyses are used for the verification of the homogeneity and stability of the test material. Especially moisture and protein parameters show small deviations and strong agreement with the reference method, which indicates good repeatability and stability. Most parameters are within acceptance criteria, besides hectoliter weight measurements, which show systematic bias. According to the findings, near-infrared (NIR) spectroscopy is a rapid, non-destructive, and reliable approach in terms of quality assessment, and its implications include improving decision-making efficiency and lowering operational costs in the grain supply chain.

Introduction

In the agricultural industry, near-infrared spectroscopy is widely used to analyze samples for their compositional or characteristic traits. It is a well-known analytical technique due to its advantages such as rapid, non-destructive, and multi-parameter measurement capabilities.

Recent studies have mentioned that NIR spectroscopy is one of the best analytical tools for wheat quality evaluation due to its ability to enable rapid, efficient, and non-destructive analysis of multiple parameters simultaneously [9]. This study differs from related work [9], due to its originality in combining statistical measurement validation with the operational implications of NIR-based grain quality analysis. It is also important to highlight the integration of statistical quality control and laboratory performance assessment using real-time data brings an interdisciplinary perspective.

Near-infrared spectroscopy has been named near-infrared reflectance due to the use of near-infrared light to analyze characteristics. In comparison with conventional reference methods, that require more accurate measurement systems, are time-consuming, laboratory-intensive, and require more detailed sample preparation. Additionally, it is affordable while testing many parameters at the same time.

According to the working principles of NIR spectroscopy, in the NIR spectrum, light reacts with bonds such as O-H, N-H, and C-H at different wavelengths. Caporaso et al. [14] mention that NIR-based analytical models have strong spectral sensitivity associated with O–H, C–H, and N–H molecular bonds, which enables robust prediction.

Regions with higher protein concentration absorb NIR light more strongly, resulting in lower reflected intensity in those spectral regions. The integration of NIR spectroscopy with quality control routines introduces several challenges such as measurement accuracy, calibration robustness, and long-term stability. It is obvious that the results of NIR spectroscopy not only depend on instrumentation performance but also on the quality of calibration models, the homogeneity and stability of the samples, and control of environmental conditions.
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Chadalavada et al. [11] mention that the predictive performance of NIR spectroscopy is highly affected by chemometric calibration, metrological processing, and the representativeness of calibration.

NIR measurement is applied due to its ability to provide simultaneous multiparameter evaluation with minimum sample preparation [4]. Badaró et al. [12] highlight that multiparameter assessment makes NIR spectroscopy suitable for routine quality control where analytical assessment and reproducibility are important.

This study addresses the need for conducting quantitative analysis of NIR spectroscopy for bread wheat quality parameter analysis. The focus is on repeatability, reproducibility, and systematic bias of measurement results.

The main aim of the study is to examine the application of measurement performance for operational efficiency and economic decision-making in grain trading. NIR systems have high potential for real-time process control and quick decision making in cereal production and grain trading operations [10]. Additionally, online NIR implementation improves process monitoring efficiency by enabling continuous quality assessment during storage, processing, and trading stages [13].

Test Materials Preparation

In the phase of test material selection, it is important to use a pure kind of bread wheat as the core material. To achieve a higher level of homogeneity, it is necessary to mix the selected materials. To protect selected samples from humidity, approximately 1,300 grams of material are packed with secure packaging and stored properly.

Sample preparation is a critical pre-analytical step that affects traceability and reliability of analytical results [1], and the chosen techniques also affect selectivity, sensitivity, and reproducibility of analytical measurement [2]. Failure in this stage leads also to failure in calibration, validation, as well as business decision making.

During the storage process, the temperature must be maintained within the range *20 ± 2°C*, and humidity must be kept between *50–60%*. During storage, the test materials must be protected from direct sunlight**.** Materials must be kept in the test room before the analysis because of equilibration and random changes in humidity level also affect the spectrum directly. Materials and analyses of NIR Spectroscopy for bread wheat are shown in Table 1.

Table 1: Materials and Analyses

Material	Required Analyses
Bread Wheat	Near-Infrared Spectroscopy Analyses: Moisture and protein Standard hectoliter weight Chemical Analyses: Moisture, protein, dry gluten, gluten index, falling number, Zeleny sedimentation, delayed (modified) sedimentation, ash, acidity in flour, and hardness

Homogeneity and Stability

Firstly, the homogeneity of selected test materials is checked by conducting standard hectoliter weight analysis, using standard methods such as moisture, protein, wet gluten, dry gluten, gluten index, falling number, Zeleny sedimentation, delayed (modified) sedimentation, ash, acidity in flour, and hardness.

NIR spectroscopy is one of the quick and non-destructive techniques that is used for the determination of key wheat quality parameters [3]. Statistical homogeneity is very important for valid performance evaluation [7].

Cochran's test is applied to verify the homogeneity of variances to detect whether one variance is significantly larger than the others. No significant outliers were detected, and the test materials were accepted as homogeneous.

Secondly, the stability test characteristics is evaluated by conducting standard methods such with automatic devices for the material characteristics as wet gluten, dry gluten, gluten index, falling number, Zeleny sedimentation, delayed (modified) sedimentation, ash, acidity in flour, and hardness. For moisture, protein, and hectoliter weight, stability methods are performed by using reference comparison methods, as those parameters are not checked by automatic devices. Hectoliter weight (with Nilemalitre), protein, Zeleny sedimentation, delayed (modified) sedimentation, hardness, and acidity in flour parameters have acceptance criteria, which are given below (in Eq.1).

$$|y_1 - y_2| \leq 0{,}3\sigma_{pt} + 2\sqrt{u^2(y_1) + u^2(y_2)} \quad (1)$$

, where σ_{pt} is target standard deviation and u is uncertainty.

The target sigma (σ) values used in homogeneity and stability control were determined based on the reproducibility (*%RSD)* values, as shown below in Table 2.

Table 2: Target sigma values

Analysis	Target sigma (σ_{pt})	Analysis	Target sigma (σ_{pt})
Hectoliter weight (by measurement device)	0,3	Delayed sedimentation	3,5
Standard hectoliter weight	0,4	Wet gluten	1,7
Protein (NIR)	0,25	Dry gluten	0,8
Protein (Standard)	1,4% of homogeneity mean	Gluten index	32-30% of homogeneity mean
Moisture (NIR)	0,25	Ash	0,03
Moisture (Standard)	0,15	Hardness	8
Falling number	9% of homogeneity mean (min:7)	Acidity in flour	5% of homogeneity mean + 0,003
Sedimentation	5% of homogeneity mean (min:2,0)	-	-

Results

Tables 3, 4, and 5 present the parameters of bread wheat test materials obtained using both reference laboratory methods and NIR measurements. It is formed around assigned values, performance criteria, and repeated analysis results. These tables enable the evaluation of homogeneity and stability.

Each value has its own assigned value for reference comparison, as well as a performance criterion for representation of upper and lower tolerances.

Table 3: Chemical Analyses of Bread Wheat

Chemical Analyses of Bread Wheat					
Parameter	Hectoliter weight	Moisture	Protein	Sedimentation	Delayed sedimentation
Unit	kg/hl	%	%(d.m.)	ml	ml
Assigned Value	83,4	11,05	12,70	28	30
Performance Criterion	0,4	0,16	0,32	5	6
Test 1	85,6	11,10	12,6	29	31
Test 2	85,8	11,00	12,5	29	31
Test 3	85,9	11,20	12,4	28	30
Test 4	85,4	11,10	12,7	28	31
Test 5	85,4	11,20	12,5	29	30

Table 4: Chemical Analyses of Bread Wheat (other parameters)

Chemical Analyses of Bread Wheat					
Parameter	Wet gluten	Gluten index	Falling number	Ash	Hardness
Unit	%	%	s	%(d.m.)	HI
Assigned Value	26,5	89	442	1,71	61
Performance Criterion	2,2	8	80	0,035	12
Test 1	26,0	77	466	1,61	66
Test 2	26,0	77	470	1,64	65
Test 3	26,4	78	464	1,61	64
Test 4	25,8	76	478	1,62	63
Test 5	26,2	75	476	1,62	65

According to the tables, it is obvious that most of the parameters show relatively low-level dispersion around the assigned values. Test results are considered within accepted performance criteria, which also highly supports acceptable repeatability and material homogeneity.

Furthermore, parameters such as moisture and protein show minimal variation, while sedimentation and falling number show slightly higher variability. Besides that, all results remain within the acceptance interval.

In the NIR measurement section, there were moisture and protein parameters with tighter acceptance criteria. Only minor deviations from the assigned values were observed, indicating good repeatability and calibration stability.

Table 5: Near-Infrared Spectroscopy Measurements

Near-Infrared Spectroscopy		
Parameter	Moisture	Protein
Unit	%	%(d.m.)
Assigned Value	11,05	12,70
Performance Criterion	0,25	0,25
Test 1	11,00	12,60
Test 2	11,00	12,50
Test 3	11,10	12,50
Test 4	11,10	12,40
Test 5	11,20	12,50

Cochran's test is also applied to evaluate variance of homogeneity. According to the controlled spread of the test results and the absence of possible outliers, the dataset supports the assumption that no significant variance dominates. Additionally, it confirms statistical homogeneity of the materials.

Furthermore, in the next evaluation of the same dataset, it is needed to be highlighted that key parameters remain constant in the repeated measurements, which demonstrates stability. Materials such as moisture, protein, and hectoliter weight cannot be handled by direct stability testing; that is why reference comparison is used, due to methodological appropriateness that confirms the sensitivity of those materials to environmental conditions.

The z-score results for chemical analyses are presented in Tables 6 and 7, while the z-score results for NIR measurements are presented in Table 8.

Table 6: Z-Score Calculation for Chemical Analyses of Bread Wheat

Z-Score Calculation for Chemical Analyses of Bread Wheat					
Parameter	Hectoliter	Moisture*	Protein*	Sedimentation*	Delayed sedimentation
Unit	kg/hl	%	%(d.m.)	ml	ml
Test 1	5,50	0,24	-0,60	0,18	0,17
Test 2	6,00	-0,24	-0,60	0,18	0,17
Test 3	6,25	0,73	-0,63	0,00	0,00
Test 4	5,00	0,24	-0,58	0,00	0,17
Test 5	5,00	0,73	-0,60	0,18	0,00

Table 7: Z-Score Calculation for Chemical Analyses of Bread Wheat (other parameters)

Z-Score Calculation for Chemical Analyses of Bread Wheat					
Parameter	Wet gluten	Gluten index	Falling number*	Ash*	Hardness*
Unit	%	%	s	%(d.m.)	HI
Test 1	-0,23	-1,50	0,28	-1,81	0,39
Test 2	-0,23	-1,50	0,33	-1,43	0,31
Test 3	-0,05	-1,38	0,26	-1,81	0,23
Test 4	-0,32	-1,63	0,42	-1,84	0,16
Test 5	-0,14	-1,75	0,40	-1,84	0,31

Table 8: Z-Score Calculation for Near-Infrared Spectroscopy Measurement

Z-Score Calculation for Near-Infrared Spectroscopy		
Parameter	Moisture	Protein
Unit	%	%(d.m.)
Test 1	-0,18	-0,37
Test 2	-0,18	-0,74
Test 3	0,18	-0,74
Test 4	0,18	-1,11
Test 5	0,53	-0,74

Z scores are used to assess laboratory performance by comparing test results with the assigned value [7]. According to the performance statistics calculations, z-scores are calculated by using the formula given below.

$$z = \frac{x-X}{\sigma} \quad (2)$$

, where x is each individual test result, X is the assigned value, and σ is the standard deviation.

Results between $-2 \le z \le 2$, are considered acceptable, while $2 < |z| < 3$ is a warning, and $z \ge 3$ is out of control [7].

For all parameters, z-scores are within acceptable criteria; only the results of hectoliter weight are exceeding ±3, which indicates systematic deviations from the assigned value. The consistency of these deviations in all measurement results of the hectoliter parameter results from method bias, not random effects. Possible reasons include calibration offset, differences in reference methods, or underestimation of target sigma values. Calibration models in NIR spectroscopy require continuous validation and maintenance to ensure long-term robustness and accuracy [6].

According to the performance test dataset, there is a bridge between analytical performance and economic value in the grain supply chain. Laboratory measurement performance is closely linked to commercial trust.

This is consistent with the role of standardized and transparent data reporting, which supports market access and reduces information asymmetry in agricultural supply chains [8].

Firstly, measurement results are within acceptance criteria, and z-scores are considered acceptable, which results in stable and reproducible measurements within the laboratory. Parameters that are checked in the chemical analysis as well as NIR measurements are directly linked with wheat classification and end-use quality. It is obvious from the measurement results that they imply low analytical uncertainty.

From a business perspective, these factors support reliable grading, reduction in disputes between buyers and sellers, and stable product specifications for related processes such as mills and bakeries.

From the business perspective, one of the critical findings is that hectoliter weight shows deviations across all measurements. Most likely, it is due to method bias such as calibration or volume determination issues, but not because of random variability. In grain trading, one of the important pricing parameters is hectoliter weight; that is why overestimating or underestimating results in pricing errors. It also affects inventory valuation, procurement costs, and contractual fairness. If it is consistently biased, it may distort market competitiveness and create financial exposure.

In Figure 1, box plots of each parameter indicate distribution symmetry and are used for group comparison.

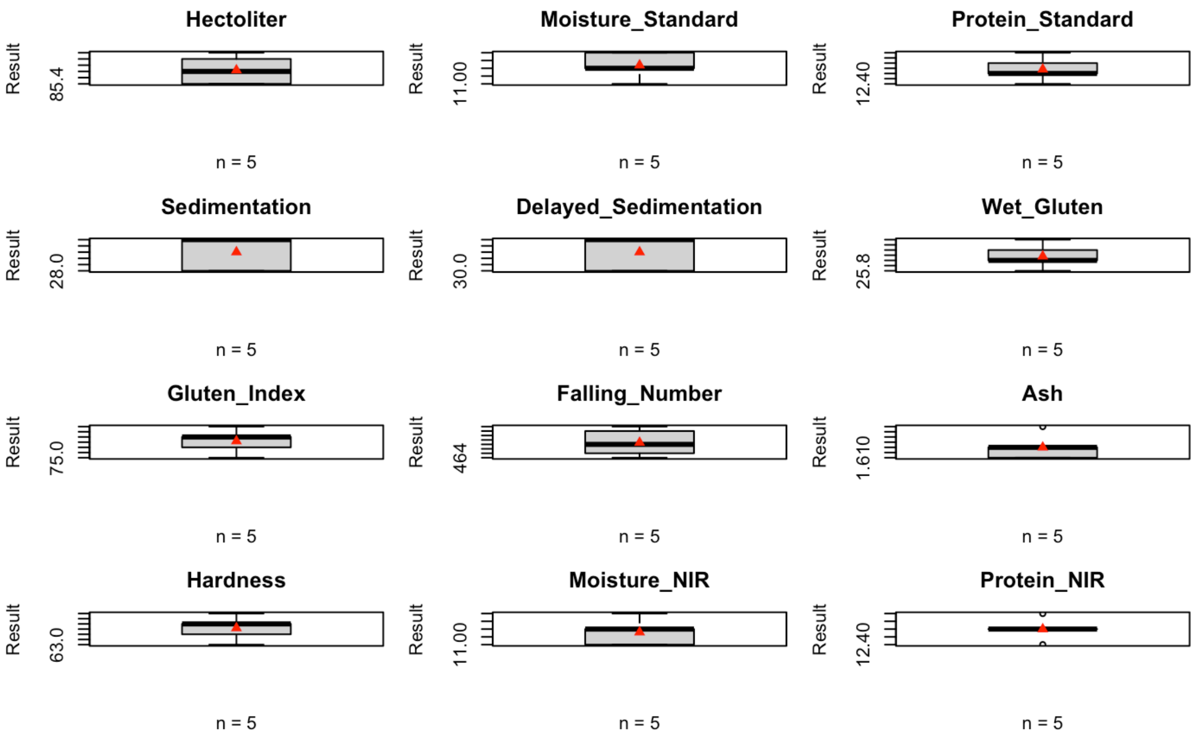

Figure 1. Box Plots of Each Parameter’s Measurement Result

Protein and moisture measurements show low variability, indicating strong method control and commercial stability. Following this, sedimentation, falling number, and hardness show higher variability, which provides greater uncertainty for predicting performance evaluation.

These factors affect risk management and production planning. Falling number affects the estimation of activities, which results in changes in flour blending decisions or material consistency.

According to the reference methods and NIR measurement methods, there is a new business-related dimension: speed of decision-making and accuracy. Muhammad Faisal Manzoor et al. describe NIR techniques as rapid analytical methods that support quick decision-making [5].

For NIR measurement results, there is low dispersion in the acceptance rate, and it supports rapid decision-making. From an operational perspective, it provides faster intake decisions and trade points, which reduces process time and logistic costs and supports optimization of resource allocation [5].

However, relying only on NIR measurements and not on reference methods can cause systematic drift, and it leads to economic losses, especially for important parameters such as protein and moisture.

In Figure 2, histograms visualize the distribution of key quality parameters of bread wheat and NIR-based measurements. Each histogram is about Test 1 to Test 5 results of each parameter and illustrates the dispersion of measurements.

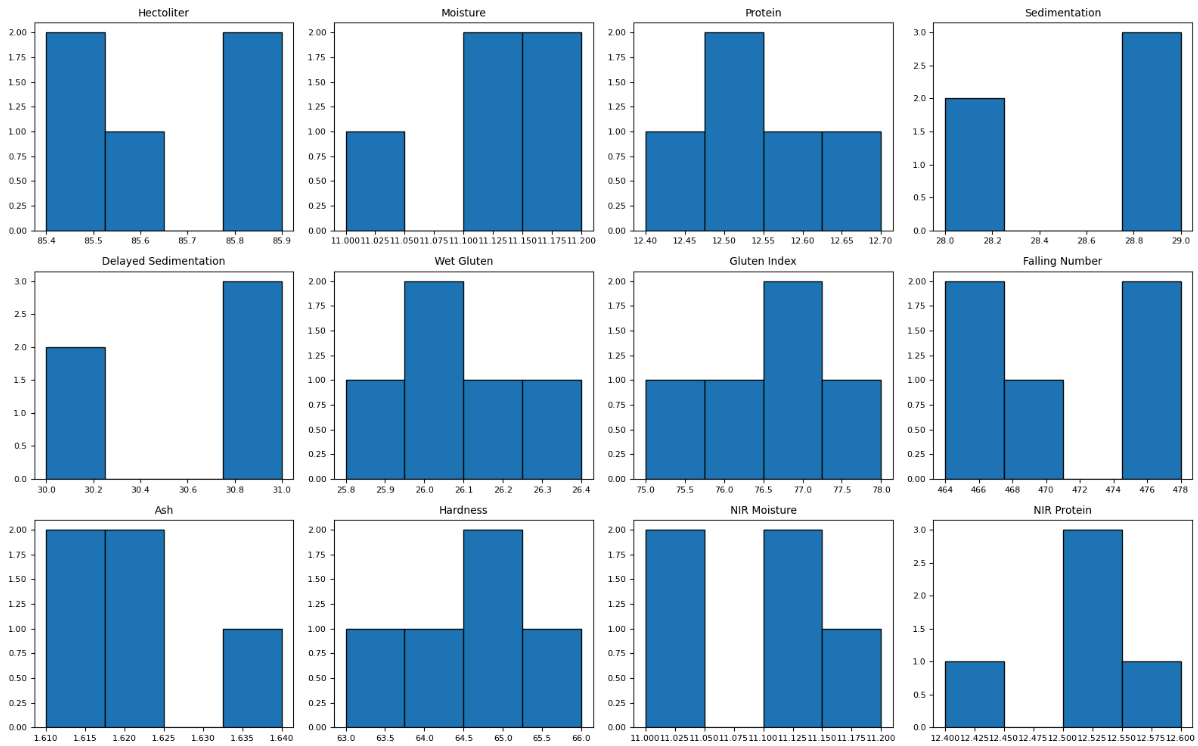

Figure 2. Histogram-Based Distribution Analysis of Chemical and Near-Infrared Spectroscopy Parameters

The observed variability is associated with test performance, not material inconsistency, as both homogeneity and stability tests support those findings. It is essential from business approach knowing that deviations are from measurement process capability, not because of material variabilities. There is a need for corrective actions to improve the reliability of processes.

Z-score ranges support comparability between measurement systems, which reduce information asymmetry over the supply chain, which is important from a business relationship perspective. Trustworthiness between producers, traders, storage operators, and processors is highly linked with these facts. In summary, such deviations disrupt this harmonization and require corrective actions to improve results for market integrity.

Overall, the dataset and the demonstrated process play a role not only as quality indicators but also as key drivers of pricing accuracy, operational trust and efficiency, and risk control in the grain industry.

Conclusion

The results obtained using both approaches demonstrate that reference methods and near-infrared spectroscopy provide reproducible measurements for evaluating bread wheat quality parameters. The samples fulfilled the requirements for homogeneity and stability, as confirmed by statistical assessment and variance analysis.

Low dispersion was observed across several measured parameters, and the results showed strong agreement with the assigned values. In particular, the moisture and protein results confirmed the repeatability and reproducibility of the analytical procedures.

A systematic bias was identified in hectoliter weight measurements, most likely reflecting methodological or calibration differences rather than random variability. If left uncorrected, this bias may cause economic distortion, systematic drift, and related risks in grain trading.

From an operational perspective, near-infrared spectroscopy reduces the need for unnecessary analyses and supports real-time decision-making in grain-industry operations. Furthermore, it improves operational efficiency, lowers costs, and facilitates resource allocation with minimal sample preparation.

The business relevance of NIR spectroscopy lies primarily in its ability to enable rapid quality verification, support standardized grading practices, reduce transaction uncertainty, and improve the accuracy of agricultural commodity valuation.

Overall, near-infrared spectroscopy can be regarded as an effective complementary tool that provides both analytical reliability and economic value. When applied correctly and supported by robust calibration and validation, NIR spectroscopy can strengthen quality-control systems and enhance transparency, pricing accuracy, and trust across the agricultural supply chain.

Data Availability

The datasets analyzed during the current study are not publicly available due to confidentiality agreements with Agro Bitki Klinikası LLC but may be available from the corresponding author upon reasonable request and with permission from Agro Bitki Klinikası LLC.

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