Annals of Agricultural & Crop Sciences

Open Access

Austin Publishing Group

Research Article

NIR Validation and Calibration of Van Soest cell wall constituents (ADF, NDF, and ADL) of Available Corn Silage in Bangladesh

Manika Debnath[1]*, Abu Sayeed[1], Abdul Hannan[1] and Abrham Ayele[2]

[1]Establishment of Quality Control Laboratory for Livestock Production Inputs and it's Food Products, Department of Livestock Services, Bangladesh

[2]University of Gondar, College of Veterinary Medicine and Animal Sciences, Bangladesh

***Corresponding author:** Manika Debnath, Senior Scientific Officer at Establishment of Quality Control Laboratory for Livestock Production Inputs and it's Food Products, Department of Livestock Services, Bangladesh

Received: June 04, 2022; **Accepted:** July 01, 2022; **Published:** July 08, 2022

Abstract

This study was undertaken at the Feed Quality Control Laboratory, DLS, Savar, Dhaka, to calibrate and validate corn silage nutritional parameters-Van Soest cell wall constituents (ADF, NDF, and ADL) in the Near-infrared Spectrophotometer (Bruker-MPA, Germany) systems monochromator (700-9500 nm) range was used for the rapid analysis of available corn silage. Almost 52 samples were analyzed at the QC lab wet chemistry laboratory to know the available nutrients. In the 2nd part of this study, developed local calibration equations in the NIRS using OPUS (Optical User Software) to relate the spectral data and corresponding wet chemistry values. A Quartz sample cup was used to hold the sample on the Infrared light scanner and used XPM was MPAII sphere macrosample_64_rotating_Res16-DLS.XPM. Fresh samples were dried and ground through 2mm screen for the analysis. The value for each component was placed into the calibration group for NIRS equation development. After calibration in NIR, the root means a square error of estimation (RMSEE) for the determination of ADF%, ADL%, and NDF%, were, 1.56, 0.47, and 1.15, with the correlation coefficient (r2) of 93.31, 95.43, and 97.86 respectively which are very close to the mean laboratory values. Whereas after cross-validation, RMSECV (Root Mean Square Error Cross-Validation) were 1.97, 0.598, and 1.55, along with the r2 values 87.02%, 90.23%, and 95.32%, respectively. The accuracy% of the predicted values in NIRS was between 98.94-100.16% which are very close to the mean laboratory values. It can be concluded that NIR could be a potential instrument to predict the nutritional quality of corn silage in Bangladesh.

Keywords: FT-NIR; Cross-Validation; Calibration; Corn Slage

Introduction

Silage is a process of forage conservation in an anaerobic environment, causing the breakdown of proteins and amino acids and subsequent production of several nitrogen compounds like amines and ammonia through some chemical reaction by plant or microbiological enzymes present in this [1]. Among all of the fodder silage, the popularity of maize silage is high, and therefore the production of maize in Bangladesh is increasing. For example, about 24.45 Lac Metric Ton maize was produced in the 2015-16 fiscal year, whereas it was 35.69 Lac Metric Ton in the 2019-20 fiscal year, increasing at 45.97% [2]. It means green corn fodder is available with the increase of corn cultivation. This availability of corn fodder (major) has helped the entrepreneur to develop business-related silage production and marketing. Along with promoting the production of fodder silage, it is imperative to maintain and monitor its quality at the farm and market level. Near Infra Reflectance Spectrophotometry (NIRS) is well known rapid screening equipment in the world. It is very much popular in the feed industry and can evaluate feed samples within a few seconds. Near-infrared spectroscopy is routinely used for the prediction of fiber concentration in forages and has greatly increased the ease of obtaining fiber analysis of forage samples. However, in ruminant nutrition, forage cellulose, hemicellulose, and lignin concentrations are commonly estimated as ADF, ADL, and NDF respectively (Van Soest and Robertson 1980) are not commonly analyzed through NIR. This study has been conducted to calibrate the FT-NIR against the wet chemistry values for evaluating three nutritional values (ADF %, ADL%, and NDF %) of available corn silage in Bangladesh. The validation in the FT-NIR-MPAII-Advanced was conducted through calibration and validation of the analytical values (Table 1) with the OPAS Lab software. Several statistical parameters are used to develop the calibration model in NIRS. Correlation coefficient (r2) RMSECV (Root Mean Square Error Cross-Validation), root means a square error of estimation (RMSEE), etc. are the indicator of linear validation. J. B. REEVES reported that the ADF and NDF predicted Coefficient of variation for the calibration of corn silage were 0.84 and 0.86 & RMSEE were 2.82 in both, whereas, in the case of Validation the predicted Coefficient of variation were 0.79 and 0.90 with RMSECV 2.85, and 3.48 in his experiments [3]. However, [4] Marten found an RDP value of 3.4 for cross-validation of corn silage in NIR. In Bangladesh, the research on NIRS for the prediction of the nutritional value of corn silage is not available, although the NIRS concept is not new in the world. Therefore, the present investigation was performed to estimate the Van Soest component (ADF, NDF, and ADL) of available corn silage in Bangladesh by NIRS and find out the importance of using

Citation: Debnath M, Sayeed A, Hannan A and Ayele A. NIR Validation and Calibration of Van Soest cell wall constituents (ADF, NDF, and ADL) of Available Corn Silage in Bangladesh. Ann Agric Crop Sci. 2022; 7(3): 1114.

Table 1: Laboratory analytical values (DM basis), NIRS values, and the relevant NIRS statistics of Corn silage.

SL	Analytes	No. of Samples	Laboratory Values %	NIRS Values (%) (Predicted)		Accuracy (%)	Cross-Validation Statistics					Calibration Statistics		
			Mean	Mean	Date range %	(98-101)	Rank	r2	RMSECV	Bias	RPD	r2	RMSEE	RPD
1	ADF%	51	31.048	30.983	22.71-42.94	99.79	7	87.02	1.97	0.0651	2.78	93.31	1.56	3.87
2	ADL%	51	6.137	6.103	3.294-11.02	99.44	7	90.23	0.598	0.0129	3.2	95.43	0.47	4.68
3	NDF%	51	50.711	50.77	38.87-64.77	100.12	7	95.32	1.55	-0.0597	4.62	97.86	1.15	6.83

RMSECV = Root Mean Square Error Cross-Validation, RMSEE = Root Mean Square Error of Estimation, r2 = Correlation coefficient, RPD= Relative percentage difference. ADF= Acid Detergent Fiber, ADL= Acid detergent Lignin, NDF= Neutral Detergent fiber.

NIRS in predicting the nutrients component in corn silage rapidly in Bangladesh.

Materials and Methods

Experimental Place and Date

The research was conducted at the Feed Quality Control Section, QC Laboratory, Savar, DLS, Dhaka, Bangladesh from July 2021 to February 2022 with the approval of the Project-Establishment of Quality Control Laboratory for Livestock Production Inputs and its Food Products, Department of Livestock Services, Bangladesh.

Sample Collection and Preparation

During sample collection, a total of 52 corn silage samples with an average weight of 4-5 kg were collected from each pit maintaining the sampling technique (ISO 6497-2002). Corn silage samples were sealed in a plastic bag or vacuum-packed and sent to the laboratory as soon as possible to reduce spoilage and also maintain temperature to avoid exposure of the sample to high temperature. One part of each fresh corn silage sample was dried immediately dried in an air forced oven at 65°C for 24 hours and ground with a 1mm sieve for the analysis.

Laboratory Analysis

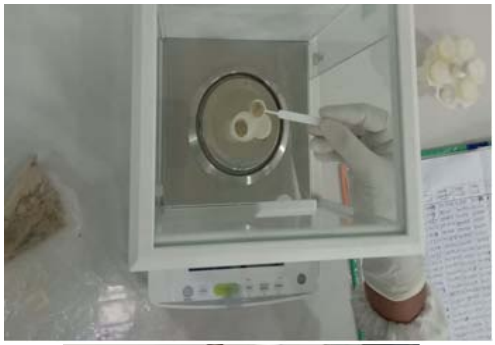
Evaluation of ADF, ADL, and NDF% in corn silage: The crude fiber/NFE system does not provide an accurate picture of the carbohydrate fraction of feedstuffs, primarily because of solubilization of variable amounts of Cellulose, hemicellulose and lignin in the crude fiber analysis. P. J. Van Soest and associates developed a rapid technique of separating feed carbohydrates on the basis of nutritional availability to ruminants and ruminal bacteria. Essentially, the method divides feeds into two fractions: (1) plant cell contents, a highly digestible fraction consisting of sugars, starches, soluble protein, pectin, and lipids; (2) plant cell wall constituents, a fraction of variable digestibility consisting of insoluble protein, hemicellulose, cellulose, lignin, and bound nitrogen. The method involves boiling a sample in a neutral detergent solution. The soluble fraction is termed neutral detergent solubles (cell contents), whereas the fibrous residue is called neutral detergent fiber (cell wall constituents). The 1g dry silage sample was boiled with an acid detergent solution. The loss in weight was resulting from the incineration of the dried residue corresponding to the weight of ADF. For the determination of ADL, additional boiling with concentrated sulphuric acid was performed before measuring the loss in weight resulting from incineration of the dried residue. Whereas, for NDF 1g dry silage sample was taken and boiled with the neutral detergent solution (NDS). The loss in weight was resulting from the incineration of the dried residue corresponding to the weight of NDF. All three analyses were conducted using the manual filtration technique.

Spectroscopic Analysis

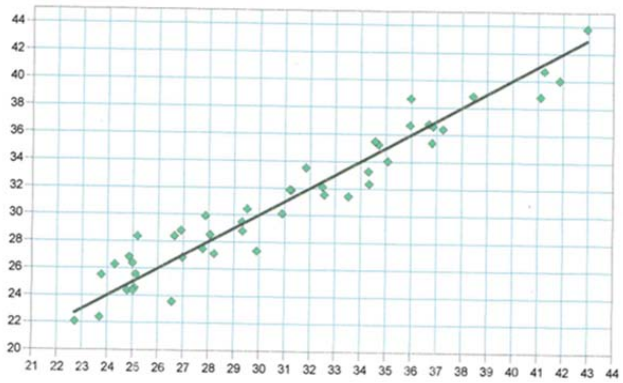
Calibration and Validation procedure: For the development of an ideal database of corn silage, NIRS- Advanced (Bruker, MPAII, Germany) systems monochromator (700-9500 nm) range was used and compared with the values evaluated from the wet chemistry analysis. A Quartz sample cup was used to hold the sample on the Infrared light scanner and used XPM was MPAII sphere macrosample_64_rotating_Res16-DLS.XPM to scan the samples. Then spectrum was stored in to a respective folder. Then the analytical data were incorporated against the spectrum to develop a calibration equation and model. The model was developed using PLS algorithm and processed the spectral data using a suitable mathematical process- derivatives, vector normalization, subtraction of straight line, and the frequency range. The cross-validation includes removing sample which was outliers, analyzing, recovering of removed sample in relation to the acceleration of regression of coefficient and RPD, and it was continued until the acceptable range was not reached successfully. To verify and establish the calibration model in the NIR, the accuracy, correlation coefficient, RMSECV, and RMSEE had been calculated. All calculated data has been given in below in the tabular (Table 1) form.

Results and Discussion

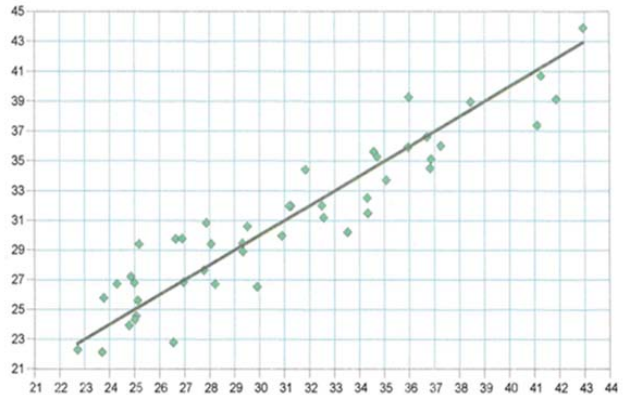
The mean predicted values of corn silage were 30.983, 6.103, and 50.77 % of ADF, ADL, and NDF% respectively. Whereas the laboratory values were very close to the NIR predicted values with 99.79, 99.44, and 100.12% accuracy. The Root Mean Square Error of Estimation (RMSEE) was calculated to optimize the calibration model indicating the relationship between analytical values and NIR predicted values. J. B. REEVES found 2.82 RMSEE for the prediction of both ADF% and NDF% in corn silage, whereas, only 1.56 and 1.15 were found in this experiment, indicating better validation. Furthermore, the RMSECV for ADF% and NDF% were 1.97 and 1.55 are less than RMSECV 2.85 & 3.84 respectively [3]. In addition, the Correlation coefficient only for ADF% was less than 90, and for other parameters such as ADL% and NDF%, were 90.23 and 95.32 are a good indication prediction after cross-validation. On the other hand, the observed r2 value in the calibration of ADF%, ADL%, and NDF% components were higher -93.31, 95.43, and 97.86 respectively. Whereas, [4] observed only 93.2 (Mårten Hetta) for NDF%. Marten also found RDP value of 3.4 for cross-validation of corn silage in NIR. According to his opinion, more than 3 RPD represent acceptable to good models. In relation to this figure, the predicted RPD values of the calibration model were more than 3 in this experiment. With ranking 7, r2 values in both calibration and cross-validation were up to the



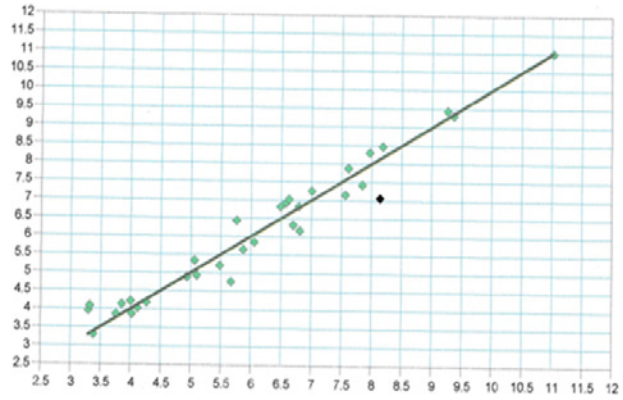
Picture: Manual filtration unit.



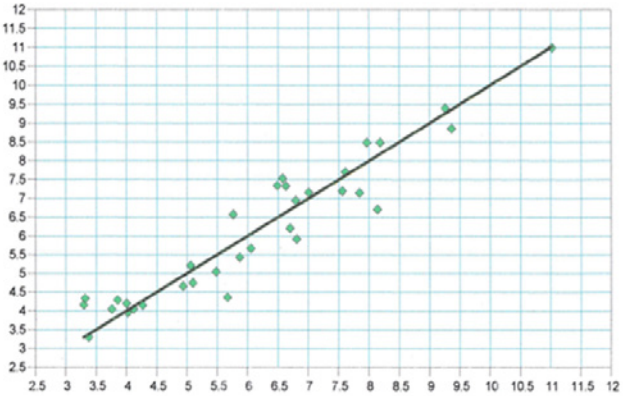
Fit vs True / ADF [%] / Calibration.
Rank: 7, R^2=93.31, RMSEE=1.56, RPD: 3.87, Validation No 3+, Corn silage ADF%.q2



Prediction vs True / ADF [%] / Cross Validation.
Rank: 7, R^2=87.02, RMSECV=1.97, Bias: -0.0651, RPD: 2.78, Validation No 3, Corn silage ADF%.q2



Fit vs True / ADL [%] / Calibration.
Rank: 7, R^2=95.43, RMSEE=0.47, RPD: 4.68, Validation No 21+, Corn Silage ADL%.q2



Prediction vs True / ADL [%] / Cross Validation.
Rank: 7, R^2=90.23, RMSECV=0.598, Bias: 0.0129, RPD: 3.2, Validation No 21+, Corn Silage ADL%.q2

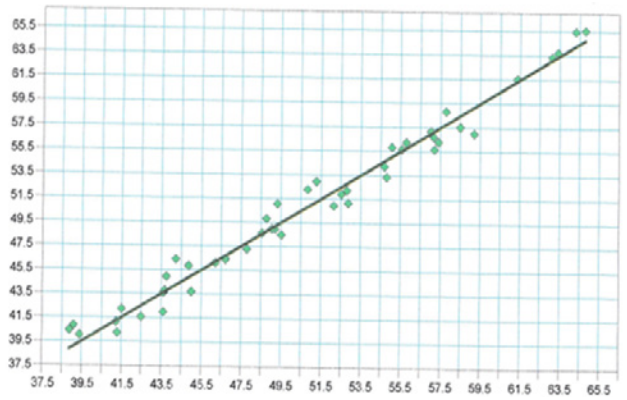
mark. However, in each component, bias was less than 1 indicating the accuracy of the model. The spectral data and the concentration data were first encoded in a matrix form. Few factors were chosen to avoid the smaller spectral changes in the data set that may create overfitting (noise). Therefore, the appropriate rank, factors with RMSECV were optimized by cross-validation to get the optimum prediction of each component.

Conclusion

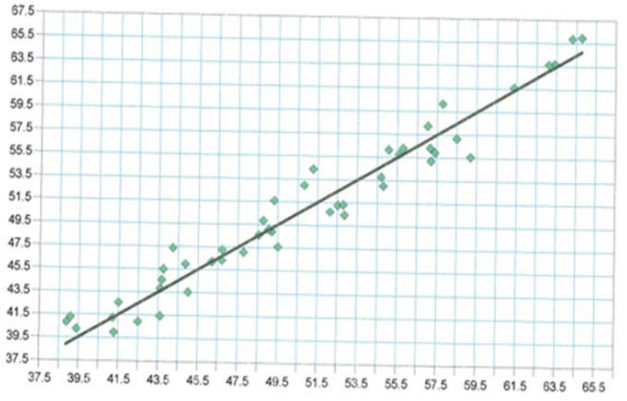
The accuracy of mean predicted values by FT-NIR and wet chemistry values of ADF%, NDF%, and ADL% of dried corn silage are very much close. Therefore, NIRS could significantly be used to predict the nutritional quality of corn silage.

Limitation

This research project evaluated only 52 samples of dry corn silage to develop this calibration model in NIR was limited extend. It is recommended that further research with a numerous number of samples may upgrade the database of Corn silage in NIR.



Fit vs True / NDF [%] / Calibration.
Rank: 7, R^2=97.86, RMSEE=1.15, RPD: 6.83, Validation No 5++, Corn Silage NDF%.q2



Prediction vs True / NDF [%] / Cross Validation.
Rank: 7, R^2=95.32, RMSECV=1.55, Bias: -0.597, RPD: 4.62, Validation No 5+, Corn Silage NDF%.q2

Acknowledgments

Investigator are grateful to the Project Director, Establishment of Quality Control Laboratory for Livestock, Production Inputs, and its Food Products, Department of Livestock Services for providing funds and opportunities to conduct this research work at the QC Laboratory, DLS, Savar, Dhaka. Finally, special thanks are given to the staff of the Feed Quality Control Section for their cordial assistance.

References

1. Mitsuaki Ohshima, Peter McDonald. A review of changes in nitrogenous compounds of herbage during ensilage. J Sci Food Agric. 1978; 29(6): 497-505.
2. BBS, Bangladesh Bureau of Statistics-Government of the People\'s Republic of Bangladesh, 2020.
3. I. a. T. H. B. J. B. REEVES. Near-Infrared Reflectance Spectroscopy for Analyzing Undried Silage1. Beltsville Agricultural Research Center, United States Department of Agriculture.
4. Mårten Hetta, Zohaib Mussadiq, Johanna Wallsten, Magnus Halling, Christian Swensson, et al. Prediction of nutritive values, morphology and agronomic characteristics in forage maize using two applications of NIRS spectrometry. Acta Agriculturae Scandinavica, Section B — Soil & Plant. 2017; 67: 326-333.