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Development of Local Calibrations and Validation for the Prediction of Nutrients Content in Animal Feeds Using Different Wavelength Range of Near Infrared Reflectance Spectroscopy

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Abstract

Commercial livestock and poultry farming is one of the fastest growing and most promising industries in Bangladesh. Livestock industry depends solely on compound feeds which represents 65 - 70% of the total cost of production resulting needs proper attention to evaluate the nutritional quality of feeds quickly. The aim of this study was to develop local calibration procedures using NIRS with different wavelength ranges and validation of calibrations for the prediction of nutrients in animal feeds. Five NIRS machines having different wavelength ranges of 400 - 2500 nm (DS2500 monochromator, dispersive), 800 - 2500 nm (MPA, Matrix F and Antaris, fourier transform) and 920 - 1678 nm (VIAVI MicroNIR, portable type) were selected for the spectroscopic measurement of the animal feed samples. Multivariate analyses were performed to develop calibration equations of nutrients and data were centered using Partial Least Squares (PLS) algorithm and spectral outliers were identified from each calibration. The accuracy of the calibration models were validated by root mean square error cross validation (RMSECV), ratio of performance to deviation (RPD) and correlation coefficient (R^2) between the Aunir reference and laboratory values vs predicted values of NIRS. The square error cross validation (SECV) for the evaluation of moisture (0.46 - 0.50%), protein (1.16 - 1.37%), fat (0.40 - 0.53%), fibre (0.69 - 0.75%) and ash (1.28 - 1.86%) in animal feeds by using 400 - 2500 and 800 - 2500 nm indicating higher potentiality of the models. Similarly, the RPD values (> 2.5) and R^2 (moisture > 0.88; protein > 0.98; fat > 0.86; fibre > 0.96 and ash > 0.85) were proved the accuracy of the model using 400 - 2500 and 800 - 2500 nm. In MicroNIR with wavelength range of 920 - 1678 nm, the SECV for the prediction of protein (2.18), fibre (1.13) and ash (2.32) were relatively higher than wavelength ranges of 400 - 2500 and 800 - 2500 nm. Besides, the RPD value of predicting fat contents by MicroNIR (920 - 1678 nm) was < 2 considered to not give a relevent prediction of the fat content in animal feeds. Therefore, the present study revealed that using NIRS with 400 - 2500 and 800 - 2500 nm wavelength could potentially used in predicting nutrient contents in animal feeds. However, the encouraging results obtained in this study by MicroNIR (920 - 1678 nm) suggest that by expanding number of samples in calibration and collection a higher reliability, quality and predictive capability of the models could be used for the prompt evaluation of animal feeds.

Keywords: *Nutrient Content; Nutrient Evaluation; Animal Feeds; NIRS; Wavelength Range*

Introduction

Commercial livestock and poultry farming is one of the fastest growing and most promising industries in Bangladesh. Intensive production system, however, depends solely on compound feeds which represents about 65-70% of the total cost of animal production. However, proper attention should be given to evaluate the nutritional quality of feeds and ingredients in order to supply the adequate amount of balanced diet for maximizing the productivity cost effectively. Bangladesh is going to develop nuclear based techniques in evaluating animal feeds and forages for improving poultry and dairy production. In the mean time, the Department of Livestock Services (DLS) in Bangladesh formulated the Animal Feed Act, 2010 and The Animal Feed Rules, 2013 for the quality control of animal feeds. There are about 6.0 million tons of commercial feeds have been produced by the feed millers annually [1]. Until recently, the exchange of value between buyers and sellers of feed grain in the different commercial feed millers in Bangladesh hasn't been nearly so transparent. With no proven, dependable way to demonstrate the feeding quality of grains in livestock rations at point of sale, grain growers and livestock producers have based their transactions on simple physical measurements such as bushel weight or chemical analysis by wet chemistry laboratory which may takes few days or more. But this long-standing value estimation is changing, led in large part by the demand for measurements that more accurately represent animal performance and the development of a technology known as Near-Infrared Spectroscopy (NIRS).

NIRS offers the potential for obtaining a rapid, nondestructive and accurate estimate of the chemical composition of feedstuffs and forages [2]. The technique has extensive application for the analysis of constituents of agricultural crops, feeds and foods [3-5]. This analytical tools requires no chemical reagents, therefore, avoids the problems of organic and other chemical waste disposal. Once calibrations are in place, it takes just minutes to have the result of one or more constituents. Currently, NIRS of whole grains at the grain elevator is used widely in the USA, Canada, Australia and Europe for evaluation of protein and moisture content of grains [6]. The NIRS method is based on the absorptions of C-H, N-H, and O-H groups present in organic constituents [7]. This absorption falling in the 700 - 2500 nm [7], are essentially the overtones and combination bands of the stronger ones found in infrared zones [8]. Several studies have dealt with NIR calibrations for crude protein [9,10], fibre fractions [11] and ash contents [12]. In Bangladesh, top leading feed mill operators have been using NIRS in the decade for the determination of nutritional quality of raw materials and compound feeds [13,14]. However, in order to evaluate feeds more accurate and precisely it is essential to build calibrations by including large number of local samples into the global database. Besides this, there are different types of NIRS machines and brands having different wavelength ranges and prices available in the market. In these circumstances, it is essential to know about the potential of different brands of NIR spectroscopy machines and wavelength ranges with calibrations for the nutritional evaluation of locally available compound feeds in Bangladesh for sustainable livestock production.

Aim of the Study

The aim of this study was to develop local calibration procedures using different NIRS machines with wavelength ranges and validation of calibrations for the evaluation of compound feeds and ingredients.

Materials and Methods

Preparation of sample

About 300 animal feed samples (100 samples from Aunir reference and 200 samples from the local markets in Bangladesh): cattle feed, chicken feed, horse feed and rabit feeds were selected for the calibrations and prepared for homogenious particle size (1 mm) using Cyclotec (FOSS Tecator, Denmark) feed grinder machine and dried at 60^0 C for wet chemistry analysis. Samples were stored in the refrigerated store room.

Spectroscopic measurement of the sample

Five different NIRS machines with different wavelength ranges were selected for the spectroscopic measurement of the animal feed samples. The NIRS machines were FOSS (DS2500, wavelength ranges: 400 - 2500 nm, monochromator dispersive), Bruker (MPA, wavelength range: 800 - 2500 nm, fourier transform), Bruker (Matrix F, wavelength range: 800 - 2500 nm, fourier transform, fiber optics probe), Thermo Scientific (Antaris, wavelength range: 800 - 2500 nm, fourier transform) and portable type MicroNIR (VIAVI, wavelength range: 920 - 1678 nm).

Ground feed samples were scanned in duplicate (scanning number 32) by small quartz cup with ISIScan Nova software in DS2500. Similarly, feed samples were scaned in duplicate by using OPUS Lab software in the Bruker MPA and Bruker Matrix NIRS. In the case of portable MicroNIR the samples were scaned by the USB-powered MicroNIR pro using Live scan software. At the time of scanning, samples were packed into a Qurtz cup sample holder which holds the sample into a clear glass window and to maintain good contact between the granular sample and window. NIR light was focused on to a concave mirror which separated the light into its composing wavelengths. Required wavelengths according to different NIRS machines were selected by which the near infrared light was fallen on to the feed stuffs. The amount of this light which was reflected by the feedstuff was measured to obtain the absorption corresponding to selected wavelength. Spectral reference curves were measured each of the animal feed sample and the data were stored in selected folder. Sample moisture, crude protein (CP), crude fiber (CF), crude fat (EE), total ash, were determined by the Aunir refeence wet chemistry laboratory according to the procedure of AOAC [15].

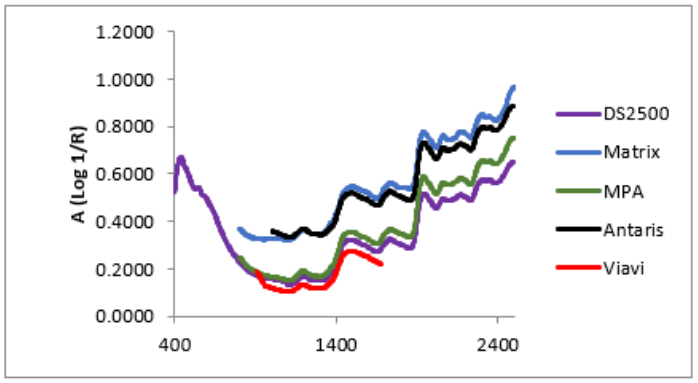

Figure 1: *Spectrum properties and wavelength range of different NIRS machines.*

Calibration and cross validation

For the transformation of spectrum, data management and the development of calibration model in the present study, three different softwares were used. WinISI was used for the development of calibration and validation of the spectrum derived from DS2500 and Antaris NIRS model and correspondence reference values of each sample was taken from Aunir reference values. The optical user software (OPUS) was used for the data management and development of calibration for Bruker MPA and Matrix spectrum of the samples. Unscrambler software was used to calibrate the spectrum from portable MicroNIR VIAVI. Multivariate analysis was performed by these commercial software to relate the spectral data and corresponding concentration values for each nutrient component moisture, protein, fat, fiber and total ash of animal feed samples [16]. In all the softwares, the model was developed using Partial Least Squares (PLS) algorithm and the spectral data were processed by a suitable mathematical method e.g. first derivative + SNV. PLS uses constituent concentration

information during spectral decomposition, which weights spectra containing higher constituent concentrations more heavily. The term "factor" or "rank" is used to describe a linear combination of spectral data. PLS reconstructs a spectrum that represents the predicted constituent values. This predicted spectrum is subtracted from the actual spectrum to determine residuals. Therefore, the residual (*Res*) is the difference between the true and fitted value. Thus the sum of squared errors (*SSE*) is the quadratic summation of these values:

$$SSE = \sum [Res_i]^2$$

The standard error of calibration (SEC) or root mean square error of estimation (*RMSEE*) is calculated from this sum, with *M* being the number of standards and *R* the PLS factors or rank:

$$RMSEE = \sqrt{\frac{1}{M-R-1}\ SSE}$$

Appropriate frequency range of the spectrum was selected to get good correlation between the changes in spectral and the concentration data. The suitability of the chosen data processing method and the frequency range for method development was evaluated during validation. In the case of cross validation, individual samples were taken from the calibration set. Using the remaining samples, a calibration model was established and used to analyze the previously extracted samples. This procedure of removing samples, analyzing them, and returning them to the calibration data set was continued successively until all the samples had been analyzed once (Figure 2).

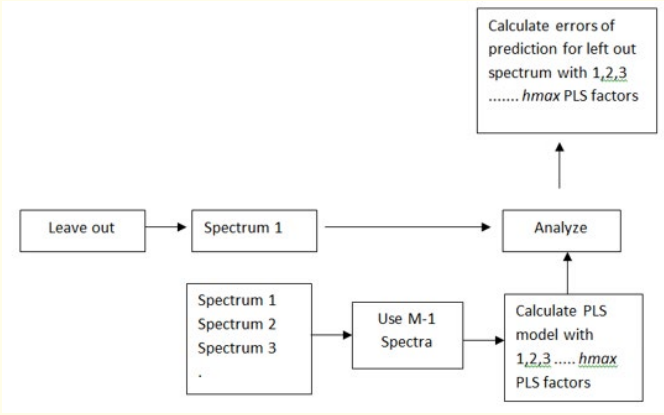

Figure 2: *Steps in cross validation (hmax is the maximum factor specified by the user).*

A comparison of the resulting analysis values with the original raw data allowed the calculation of the predictive error after cross validation of the complete data system, the root mean square error cross validation (*RMSECV*):

$$RMSECV = \sqrt{\frac{1}{M} \sum_{i=1}^{M} (Differ_i)^2}$$

Prediction of nutrients by using NIRS calibration

A separate group of samples (19 samples) without calibration set were used to predict the nutrient contents (moisture, protein, fat, fiber and total ash) of the samples by using calibrations for the different nutrients and to estimate the predictive error (RMSEP).

$$RMSEP = \sqrt{\frac{1}{M} \sum_{i=1}^{M} (y_{pred} - y_{obs})^2}$$

In order to calculate the repeatability of each nutrient predicted by using calibrations of different NIRS machines separate Excel sheet were used. Similarly, to estimate the RPD (Ratio of Performance to Deviation) following equations were used:

$$Repeatability = \sqrt{\frac{1}{M-1} \sum_{i=1}^{M} (y_i - \bar{y})^2}$$

$$RPD = \frac{(y1-y2)}{(y1+y2)/2} \times 100$$

Besides, coefficient of determination (R^2) from the linear regression of measured values of nutrient component determined by analytical laboratory versus predicted values by the NIR calibration was calculated by using Excel spreed sheet to give the accuracy of the model. During the validation, potential outliers could be detected easily only after all outliers had been removed from the calibration data set, and finally after the optimum parameters had been found, and the calibration model was established.

Results

The range of moisture, protein, fat, fiber and ash contents in animal feeds were 7.00 - 12.00, 13.70 - 48.00, 1.28 - 7.45, 3.20 - 18.90, and 6.30 - 29.70% with SD values were 1.21, 10.91, 1.44, 4.16 and 6.59, respectively (Table 1). The square error cross validation (SECV) for the prediction of moisture, protein, fat, fiber and ash contents in animal feeds by NIRS using DS2500 (400 - 2500 nm) were 0.46, 1.23, 0.53, 0.74 and 1.73% and the SEP were 0.39, 1.17, 0.59, 0.72 and 1.56%, respecively. The SD repeatability for the prediction of moisture, protein, fat, fiber and ash cntents in animal feeds using wavelength range of 400 - 2500 nm were 0.10, 0.36, 0.11, 0.25 and 0.88 with RPD of 3.18, 9.62, 2.59, 5.33 and 3.77, respectively. The correlation coefficient (R^2) between aunir reference values and NIRS predicted values were 0.90, 0.99, 0.86, 0.96 and 0.75, respectively (Table 2). Similarly, the SECV for the prediction of moisture, protein, fat, fiber and ash contents in animal feeds by using NIRS Matrix F (800 - 2500 nm) were 0.47, 1.16, 0.40, 0.75 and 1.64% and the SEP were 0.39, 0.91, 0.34, 0.74 and 1.91%, respecively. The SD repeatability for the prediction of moisture, protein, fat, fiber and ash cntents in animal feeds using wavelength range of 800 - 2500 nm were 0.04, 0.09, 0.05, 0.13 and 0.30 with RPD of 3.18, 12.36, 4.50, 5.19 and 3.08, respectively. The correlation coefficient (R^2) between aunir reference values and NIRS predicted values were 0.94, 0.99, 0.97, 0.98 and 0.94, respectively (Table 2).

Nutrients	Unit	Number of samples	Min.	Max.	Mean	SD[1]
Moisture	g/ 100g	300	7.00	12.00	9.63	1.21
Protein	g/ 100g	300	13.70	48.00	26.51	10.91
Fat	g/ 100g	300	1.28	7.45	3.78	1.44
Fiber	g/ 100g	300	3.20	18.90	7.72	4.16
Ash	g/ 100g	300	6.30	29.70	16.21	6.59

Table 1: *Descriptive statistics of nutrients in animal feeds included in NIR calibrations.*

[1]SD = Standard Deviation.

Nutrients	Number of samples in validation set	SECV[1]	SEP[2]	SD Repeatability[3]	RPD[4]	R[5]
NIRS DS2500 (Wavelength range: 400 - 2500 nm)						
Moisture	19	0.46	0.39	0.10	3.18	0.90
Protein		1.23	1.17	0.36	9.62	0.99
Fat		0.53	0.59	0.11	2.59	0.86
Fiber		0.74	0.72	0.25	5.33	0.96
Ash		1.73	1.56	0.88	3.77	0.85
NIRS Matrix F (Wavelength range: 800 - 2500 nm)						
Moisture	19	0.47	0.39	0.04	3.18	0.94
Protein		1.16	0.91	0.09	12.36	0.99
Fat		0.40	0.34	0.05	4.50	0.97
Fiber		0.75	0.74	0.13	5.19	0.98
Ash		1.64	1.91	0.30	3.08	0.94
NIRS MPA (Wavelength range: 800 - 2500 nm)						
Moisture	19	0.48	0.40	0.07	3.10	0.94
Protein		1.21	1.17	0.25	9.62	0.99
Fat		0.48	0.42	0.09	3.64	0.96
Fiber		0.69	0.93	0.24	4.13	0.97
Ash		1.28	1.50	0.21	3.92	0.97
NIRS VIAVI (Wavelength range: 920 - 1678 nm)						
Moisture	19	0.49	0.58	0.13	2.14	0.88
Protein		2.18	2.28	0.34	4.93	0.97
Fat		0.52	0.94	0.23	1.63	0.94
Fiber		1.13	1.19	0.11	3.23	0.94
Ash		2.32	2.70	0.62	2.18	0.88
NIRS Antaris (Wavelength range: 800 - 2500 nm)						
Moisture	19	0.50	0.59	0.21	2.10	0.89
Protein		1.37	1.19	0.32	9.48	0.98
Fat		0.53	0.55	0.18	2.77	0.87
Fiber		0.67	0.69	0.27	5.53	0.96
Ash		1.86	1.64	0.40	3.58	0.92

Table 2: *Cross validation statistics in predicting nutrients of animal feeds by using NIRS with different wavelength ranges.*

[1]SECV = Standard Error Cross Validation; [2]SEP = Standard Error of Prediction; [1]SD Repeatability = Standard Deviation of Repeatability; [1]RPD = Ratio to Performance to Deviation; [5]R^2 = Correlation Coefficient.

By using NIRS MPA (800 - 2500 nm), the SECV for the prediction of moisture, protein, fat, fiber and ash contents in animal feeds were 0.48, 1.21, 0.48, 0.69 and 1.28%, and the SEP were 0.40, 1.17, 0.42, 0.93 and 1.50%, respecively. The SD repeatability for the prediction of moisture, protein, fat, fiber and ash cntents in animal feeds using 800 - 2500 nm were 0.07, 0.25, 0.09, 0.24 and 0.21 with RPD of 3.10, 9.62, 3.64, 4.13 and 3.92, respectively. The correlation coefficient (R^2) between aunir reference values and NIRS predicted values were 0.94, 0.99, 0.96, 0.97 and 0.97, respectively (Table 2). Similarly, the SECV for the prediction of moisture, protein, fat, fiber and ash contents in animal feeds by using NIRS VIAVI (wavelenght range: 920 - 1678 nm) were 0.49, 2.18, 0.52, 1.13 and 2.32% and the SEP were 0.58, 2.28, 0.94, 1.19 and 2.70%, respecively. The SD repeatability for the prediction of moisture, protein, fat, fiber and ash cntents in animal feeds using VIAVI were 0.13, 0.34, 0.23, 0.11 and 0.62 with RPD of 2.14, 4.93, 1.63, 3.23 and 2.18, respectively. The correlation coefficient (R^2) between aunir reference values and NIRS predicted values were 0.88, 0.97, 0.94, 0.95, 0.88, respectively (Table 2). Besides, the SECV for the prediction of moisture, protein, fat, fiber and ash contents in animal feeds by using NIRS Antaris (800 - 2500 nm) were 0.50, 1.37, 0.53, 0.67 and 1.86% and the SEP were 0.59, 1.19, 0.55, 0.69 and 1.64%, respecively. The SD repeatability for the prediction of moisture, protein, fat, fiber and ash cntents in animal feeds using Antaris were 0.21, 0.32, 0.18, 0.27 and 0.40 with RPD of 2.10. 9.48, 2.77, 5.53 and 3.58, respectively. The correlation coefficient (R^2) between aunir reference values and NIRS predicted values were 0.79, 0.98, 0.87, 0.96 and 0.92, respectively (Table 2).

Discussion

Reliable information on the nutrient contents of animal feed materials can help manufacturers to efficiently arrange their formulations and to ensure that their products meet legitimate expectations at minimal costs. The nutrient contents of animal feeds are affected by many factors such as species, particle sizes and processing technique and thus can vary substantially. The NIR calibration and cross validation statistics by using different machines for the prediction of nutrients in animal feeds are shown in table 2. The better performance obtained by selecting validation samples in a ranked manner is in agreement with the findings of other researchers regarding the poorer calibrations afforded by random selection among a closed population of samples [17,18].

Prediction of moisture

The standard error after cross validation of local calibration equations (RMSECV) for the prediction of moisture in animal feeds by using different NIRS machines DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 0.46, 0.47, 0.48, 0.49 and 0.50, respectively (Figure 4) those are slightly higher than the SE of cross validation observed by the Cozzolino., *et al.* [19] in fish meal for prediction of moisture (0.39). The measurement of moisture by NIRS might seem to be relatively uncomplicated due to the high relative intensity or high absorptivity of the moisture O-H bands. Due to the good correlation between dry matter and moisture, NIRS also provided satisfactory predictions on the dry matter content of animal feed materials. Similarly, the standard error of prediction (SEP) for the determination of moisture in different NIR machines ranges 0.39 to 0.58 and the RPD in predicting moisture of NIR machines of DS 2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 3.18, 3.18, 3.10, 2.14 and 2.10, respectively. To assess the success of the model by NIRS, RPD values < 2 were considered to not give a relevent prediction; values between 2 to 2.5 were considered adequate for qualitative quantification; values >2.5 were considered acceptable for the prediction; and values >3 were considered that the equation could be the best for highly qualtitative analysis [20,21]. Therefore, the RPD values for the prediction of moisture by DS2500, Matrix and MPA were >3 indicating the accuracy of the model could be due to broader wavelength range of 800 - 2500 nm. Meanwhile, VIAVI and Antaris could be adequate for qualitative analysis which might be the shorter range of 920 - 1678 nm in VIAVI. Evangelista., *et al*. [22]. reported that the inability of portable NIRS to reach satisfactory prediction of nutrients appears to be related more to the instrumental resolution as well as reduced spectral range of the instruments.

Prediction of protein

The SECV for the prediction of protein content in animal feeds using different wavelength range of NIRS machines DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 1.23, 1.16, 1.21, 2.18 and 1.37, respectively those are little bit higher than the the SE of cross validation observed by the Cozzolino., *et al.* [19]. Among all NIRS machines, the SECV for the prediction of protein in VIAVI (920 - 1678 nm) is much higher (2.18) than other wavelength ranges. Cozzolino *et al.* [19] investigated the spectral characterization of protein meals and found that the bands at 1490 nm (N-H stretch first overtone), 2060 nm (carbonyl stretch of the primary amide), and 2168-2180 nm (N-H bend second overtone, C-H stretch/C=O stretch combination, C=O stretch/N-H in-plane bend/C-N stretch combination) derived from the peptide absorption of the amide group (Figure 3) and had high correlations with feed crude protein and total volatile nitrogen [23]. The best calibration model was observed in 400 - 2500 nm and 800 - 2500 nm by DS2500, Matrix and MPA NIRS due to characterize the carbonyl stretch of the primary amide by 2060 nm and N-H bend second overtone, C-H stretch/C=O stretch combination, C=O stretch/N-H in-plane bend/C-N stretch combination by 2168-2180 nm. Murray [8] reported that the most relevant segment for nitrogen was centered at 2166 nm. Meanwhile, the wavelength range of 920 - 1678 nm in VIAVI might not characterize of carbonyl stretch of the primary amide by 2060 nm and N-H bend second overtone, C-H stretch/C=O stretch combination, C=O stretch/N-H in-plane bend/C-N stretch combination by 2168-2180 nm. However, Fontaine *et al.* [24] found SE of 1.99 for the prediction of CP in fish meal by NIRS.

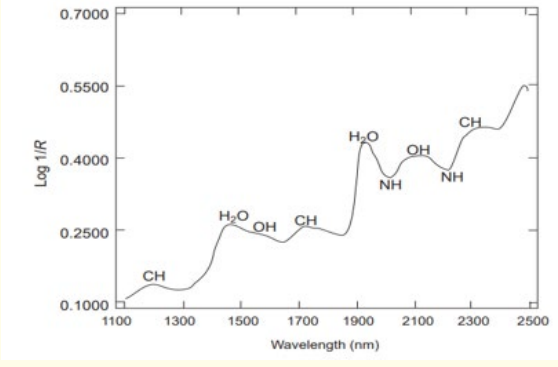

Figure 3: *NIR log 1/R spectrum showing locations of absorptions due to major functional groups.*

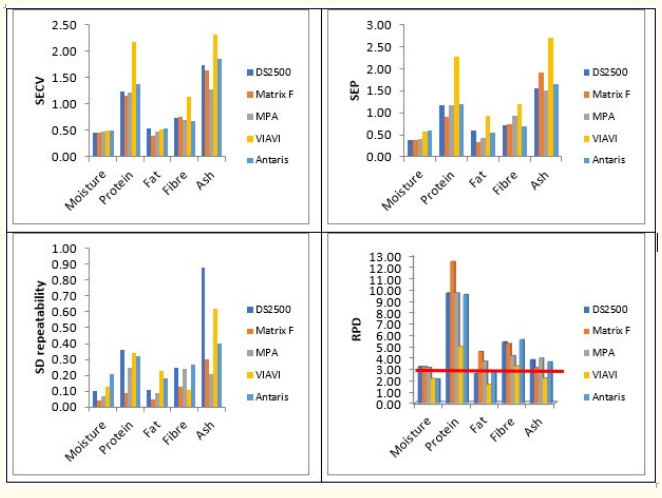

Figure 4: *Comparison of validation statistics (SECV, SEP, SDrepeatability and RPD) for the prediction of nutrients by using different NIR machines.*

Similarly, the SEP for the determination of protein in animal feeds by NIR machines of DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 1.17, 0.91, 1.17, 2.28 and 1.19, respectively indicating very good models for protein determination in animal feeds except the SEP of the model of VIAVI (2.28) was relatively higher than other models. This was due to smaller wavelength range of VIAVI (920 - 1678 nm) might not able to characterize most relevant section of nitrogen in 2166 nm. Besides, Evangelista., *et al.* [22] reported that the inability of portable NIRS to reach satisfactory prediction of nutrients appears to be related more to the instrumental resolution as well as reduced spectral range of the instruments. The RPD values of predicting protein contents in animal feeds by different NIR models were 9.62, 12.36, 9.62, 4.93 and 9.48 indicated that the equations could be used for highly accurate qualtitative analysis. However, the correlation coefficient (R^2) for the prediction of protein in animal feeds by different NIRS machines were > 0.97 indicating the high level performance of the models.

Prediction of fat

Fat is the main factor for determining energy level in the animal feed materials. Compare to moisture and protein, fat content is more difficult to be accurately predicted, especially in the compound animal feed materials. However, the calibration of lipid should be possible due to the characteristic aliphatic -CH adsorption. The SECV for the prediction of fat content in animal feeds using different wavelength range of NIRS machines DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 0.53, 0.40, 0.48, 0.52 and 0.53, respectively would much better to the SE of cross validation observed by the Cozzolino., *et al.* [19] in predicting fat contents in protein meals (0.81) and plant materials by Chen., *et al.* [25]. The SEP for the determination of fat contents in animal feeds ranges 0.34 to 0.59 in different NIR machines similar to the results by the Niu [26] who found RMSEP of fat contents in fish meal was 0.57. However, SEP for the determination of fat within the range of 920 - 1678 nm by VIAVI was relatively higher (0.94) than other machines could be due to smaller wavelength range might not able to absorp the bands responsible for fat at 2300-2500 nm. Besides, Evangelista., *et al.* [22]. reported that the inability of portable NIRS to reach satisfactory prediction of nutrients appears to be related more to the instrumental resolution as well as reduced spectral range of the instruments. Similarly, the fat content in fish meal was accurately predicted by Tan [27] with high correlation (R^2 = 0.91) and lower error (MARE = 4.53). Besides, in the present experiment, the correlation coefficient (R^2) for the prediction of fat in animal feeds by different NIRS machines were >0.86 indicating the well performance of the models. The RPD values of predicting fat contents in animal feeds by different NIR machines having wavelength ranges of 400 - 2500 nm, 800 - 2500 nm. 800 - 2500 nm, and 800 - 2500 nm were 2.59, 4.50, 3.64 and 2.77 respectively indicated that the equations could be used for highly accurate qualtitative analysis of fat in the animal feeds except the values of RPD in predicting fat contents by using VIAVI (920 - 1678 nm) of 1.63 was not satisfactory. Williams and Sobering [21] suggested the value of RPD <2 were considered to not give a relevent prediction of the nutrients.

Prediction of fibre

As in the case of protein, the SECV for the prediction of fiber in animal feeds by using different wavelength range of NIRS machines DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 0.74, 0.75, 0.69, 1.13 and 0.67, respectively in which the SE in predicting fiber by VIAVI with wavelength range of 920-1618 nm was relatively higher than other NIRS machines. This could be due to fiber contents in animal feeds was related to absorption regions of 1700-1730 nm (C-H stretch first overtone) and 2300-2310 nm (C-H bend second overtone) which might not characterize by the VIAVI NIR machine. Workman [23] reported that among the bands associated with fibre as cellulose, O-H stretch 1st and 2nd overtones, localised at 1490 and 1780 nm respectively were related to absorption. Also, he reported the 2300-2360 nm interval frequently occurring in wavelength selection for the forages fiber multivariate calibration which was similar to characterize fiber by DS2500, Matrix and MPA with 400-2500, 800-2500 and 800 - 2500 nm respectively in the present study. For the prediction of NDF as fibre, Buxton and Mertens [28] observed a 1726 nm brand close to the 1736 nm selected for the calibration model. Also, these authors reported for ADF absorption

(1560-1564 nm and 2030 nm) close to the 1576 nm and 2004 nm for the calibrations. Workman [23] match quite well with the 1682 nm for ADF calibration process which might be defined by the 920 - 1678 nm range with VIAVI NIR. Once again, Danieli., *et al.* [29] reported accuracy for NDF content as fibre with the SEP of 2.14%. In the present experiment, the SEP for the fibre prediction was 1.19 with 920 - 1678 nm range could be good enough for the calibration model. The SEP for the prediction of fibre by using 400-2500, 800 - 2500 nm ranges were 0.72 and 0.75 indicating the well performance of calibration model. The RPD values of predicting protein contents in animal feeds by different NIR machines DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 9.62, 12.36, 9.62, 4.93 and 9.48, respectively indicated that the equations could be used for highly accurate qualtitative analysis of fibre. However, the correlation coefficient (R^2) for the prediction of fibre in animal feeds by different NIRS machines were >0.95 indicating the high level performance of the models.

Prediction of ash

In predicting ash content in animal feeds, the SECV by the different wavelength ranges of DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 1.73, 1.64, 1.28, 2.32 and 1.86 respectively. Ash is the inorganic residue remaining after the water and organic matter have been removed by heating in the presence of oxidizing agents. Ash content provides a measure of the total amount of minerals in feed materials. Ash content in most feed protein materials were analyzed by NIRS with $R^2 > 0.65$ or RMSEP < 1.5% [25]. The results of the present study are somewhat surprising because the ash residue, mainly made up of mineral elements, has no characteristic NIR absorption bands. However, previous studies have proved that NIRS is capable of predicting mineral elements in plant materials [30-31]. It is proposed that NIRS can be used to evaluate mineral constituents due to the correlation between the minerals and the organic components, either through associations with organic molecules or by forming salts, affecting hydrogen bonds in samples [30]. This indirect correlation may also be responsible for good prediction results for ash content in feed protein materials. The RPD values of predicting ash contents in animal feeds by different NIR machines DS2500 (400 - 2500 nm), Matrix F (800 - 2500 nm). MPA (800 - 2500 nm), VIAVI (920 - 1678 nm) and Antaris (800 - 2500 nm) were 3.77, 3.08, 3.92, 2.18 and 3.58, respectively indicated that the equations could be used for highly accurate qualtitative analysis of fibre. Besides, the correlation coefficient (R^2) for the prediction of ash contents in animal feeds by different NIRS machines were >0.85 indicating very good performance of the models.

Conclusion

Near infrared reflectance spectroscopy may permit the reliable prediction of moisture, protein, fat, fibre and ash contents in the animal feeds within the wavelength ranges of 400 - 2500 nm (monochromator, dispersive) and 800 - 2500 nm (fourier tansform). The standard error after cross validation (SECV), standard error of prediction (SEP), correlation coefficient (R^2) and RPD values for the evaluation of moisture, protein, fat, fibre and ash contents in animal feeds by using 400 - 2500 nm and 800 - 2500 nm indicating the potentiality of the instruments. However, in MicroNIR having wavelength range of 920 - 1678 nm, SECV and SEP for the evaluation of protein, fibre and ash content in animal feeds were relatively higher than the evaluation within the ranges of 400 - 2500 nm and 800 - 2500 nm. Meanwhile, the RPD value of predicting fat contents by MicroNIR (920 - 1678 nm) was <2 was considered to not give a relevent prediction of the fat content in animal feeds. However, the encouraging results obtained in this study by MicroNIR having wavelength range of 920 - 1678 nm suggest that by expanding number of samples in calibration and collection a higher reliability, quality and predictive capability of the models in the field of animal nutrition could be achieved.

Conflict of Interest

No potential conflict of interest was reported by the author(s).

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