Report on the calibration and validation of NIRS calibration profile for proximate composition of yam flour

Overview:

Calibration models was developed for proximate and antinutrient composition of yam flour using a total of 126 genotypes obtained from the experimental fields of International Institute of Tropical Agriculture across four locations. The fresh yam tubers were processed to flour and their spectra data were collected by scanning on the benchtop near infrared spectrometer. Yam samples from the four locations were pooled to obtain the calibration set which was used to generate the models. Validation of the model was done using selected set of new materials which are not part of the calibration data set.

Sampling and measurement protocols

Materials:

2.1 Source of materials:

The yam tubers were sourced from the experimental fields of International Institute of Tropical Agriculture (IITA). A total of 126 yam genotypes were used for the calibration model development and another set of 37 genotypes were used for the validation of the models. The tubers were washed, air-dried, peeled, and again washed and dried with soft paper. Each peeled tuber was cut into four portions longitudinally from the proximal to the distal end. Two opposite portions from each tuber were pooled and homogenized. Some of the homogenized samples were put in paper bag and transferred into an oven to dry at 65°C for 72 hours. The resulting dry chips were milled to fine flour using a stainless-steel laboratory mill, then packed in a well-labelled polyethylene whirl-pack bag, and stored at 4°C until analysed in the laboratory.

PROXIMATE AND ANTINUTRIENT ANALYSIS

The samples were analysed in duplicate, for moisture, ash, protein, sugar, starch and amylose using methods described by AOAC (2005). Moisture content was determined by heating the sample in a "moisture aluminium can" to a constant weight in an oven maintained at 105 °C for 16hrs. Ash was determined by the incineration of 2 g samples placed in a muffle furnace and maintained at 550 °C for 5 hrs. Fat content was obtained by refluxing with petroleum ether in a pre-weighed extraction cups using a Soxtec apparatus, and then the final weight of the cup is taken after drying in an oven. Crude protein (% total nitrogen × 6.25) was determined by the Kjeldahl method. Phytic acid and tannin content were determined using a UV/VIS spectrophotometric method. The phytic acid was estimated by multiplying the amount of phytate phosphorus by a factor of 3.55.

Spectra collection and calibration model development.

The sample set was scanned two (2) times with wavelength range of 400 to 2498 nm, registering the absorbance values log (I/R) at 0.5 nm intervals for each sample using a NIRS monochromator (model FOSS XDS, solid module) and a stationary cell cup. Data and statistical analyses were performed using Win-ISI 4.9 software (Infrasoft International and FOSS, Hillerod, Denmark). Spectra were corrected for light scattering using the standard normal variate and de-trend (SNVD) correction. The calibration was set up using the first derivative of SNVD corrected spectra, calculated on five data points and smoothed using Savitzky–Golay polynomial smoothing on five data points. The WinISI 4.9 LOCAL regressions algorithm was used for calibration. Cross-validation with 4 groups (random) was used during calibration development. The Student (t) test was used to identify t-outlier samples during calibration development. Outlier detection was based on the standardized residuals with a cutoff of 2.5.

RESULTS:

Table 1 and 2 respectively shows the calibration and validation statistics for proximate compositions of the yam flour. Moisture, ash and fat had Mean±SD of 6.94±1.67%, 3.58±0.74% and 0.31±0.19% respectively, while protein, crude fibre and amylose had Mean±SD of 6.78±1.57%, 2.16±0.44 and 32.48±4.82% respectively. The values of sugar and starch ranged from 2.09 to 9.00% and 25.05 to 66.06% respectively, however phytate and tannin content have values in the range of 0.33 to 2.44% and 0.05 to 9.91% respectively. The coefficient of determination (RSQ) for tannin, moisture content, ash, protein, and crude fibre are 0.89, 0.87, 0.84, 0.83 and 0.80, respectively. Fat,

sugar and phytate have the lowest RSQ values. As the calibration models were validated for each constituent using new set of samples, the results showed good, high to medium, R^2 of prediction for moisture, ash, protein, and crude fibre.

Table 1: Calibration and validation statistics of proximate and antinutrient composition of yam flour

	Calibration								Validation (N=37)			
Constituent	**N**	**Mean**	**SD**	**Min**	**Max**	**SEC**	**R2cal**	**SECV**	**R2pre**	**SEP**	**Slope**	**Bias**
Moisture	126	6.94	1.67	3.79	9.75	0.56	0.87	0.68	0.80	0.54	0.95	1.36
Ash	126	3.58	0.74	1.96	6.26	0.27	0.84	0.20	0.68	0.62	1.08	0.44
Fat	126	0.31	0.19	0.05	1.73	0.14	0.07	0.20	0.14	0.28	4.25	0.49
Protein	126	6.78	1.57	3.33	9.69	0.58	0.83	0.75	0.69	0.75	0.42	0.55
Crude fibre	126	2.16	0.44	1.22	3.52	0.80	0.80	0.58	0.68	0.66	0.43	1.46
Amylose	126	32.48	4.82	18. 8	44.41	2.90	0.45	3.45	0.27	0.56	0.45	2.34
Sugar	126	5.02	1.15	2.09	9.00	0.78	0.32	0.81	0.31	0.84	0.96	1.50
Starch	126	50.68	10.32	25.05	66.06	7.05	0.54	8.02	0.42	0.88	0.72	2.46
Phytate	126	1.17	0.42	0.33	2.44	0.33	0.39	0.34	0.29	0.76	0.45	3.20
Tannin	126	2.04	1.93	0.05	9.91	0.40	0.89	0.78	0.50	0.85	0.64	2.28

SEC: standard error of calibrations; **SECV**: Standard error of cross validation;
RSQ: coefficient of determination in calibrations.

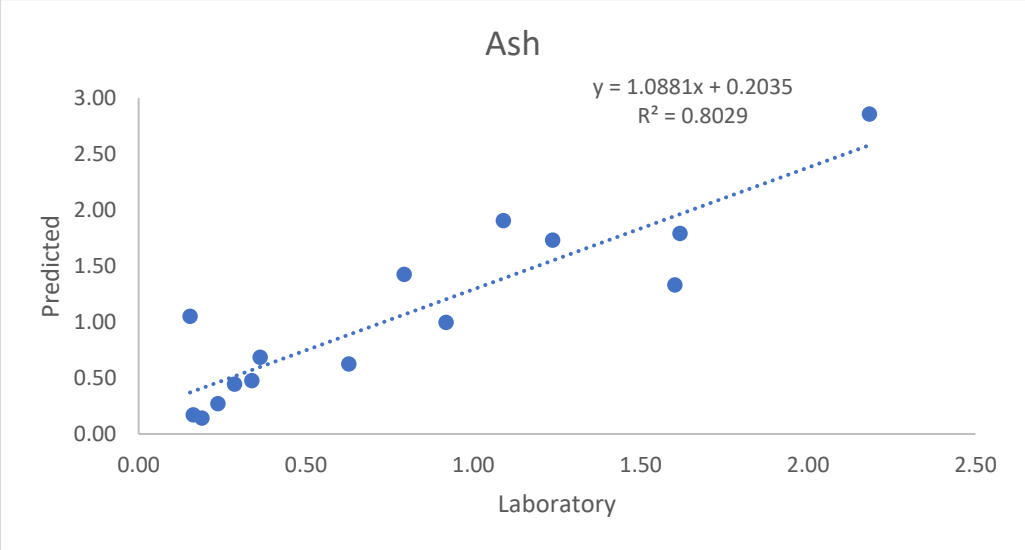

Figure 1: Graph of predicted values vs laboratory values for Ash content

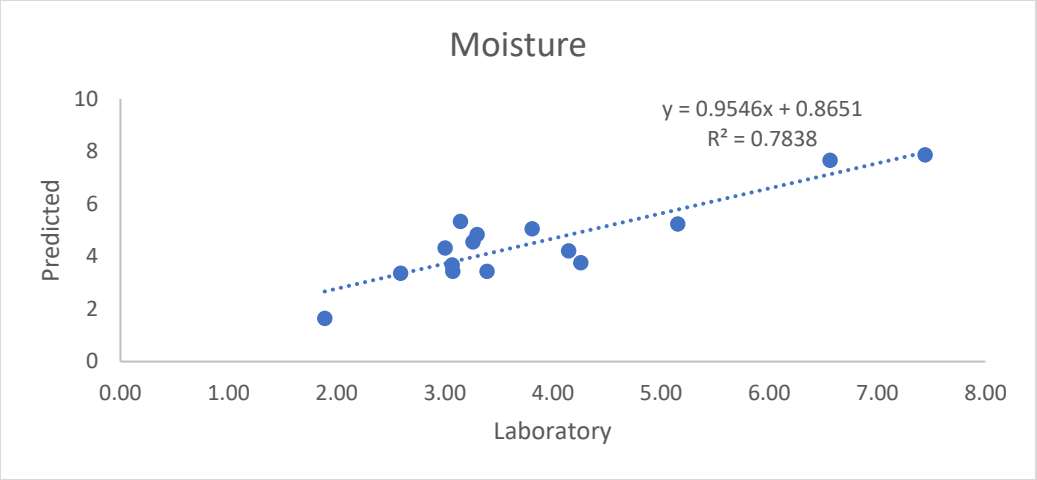

Figure 2: Graph of predicted values vs laboratory values for moisture content

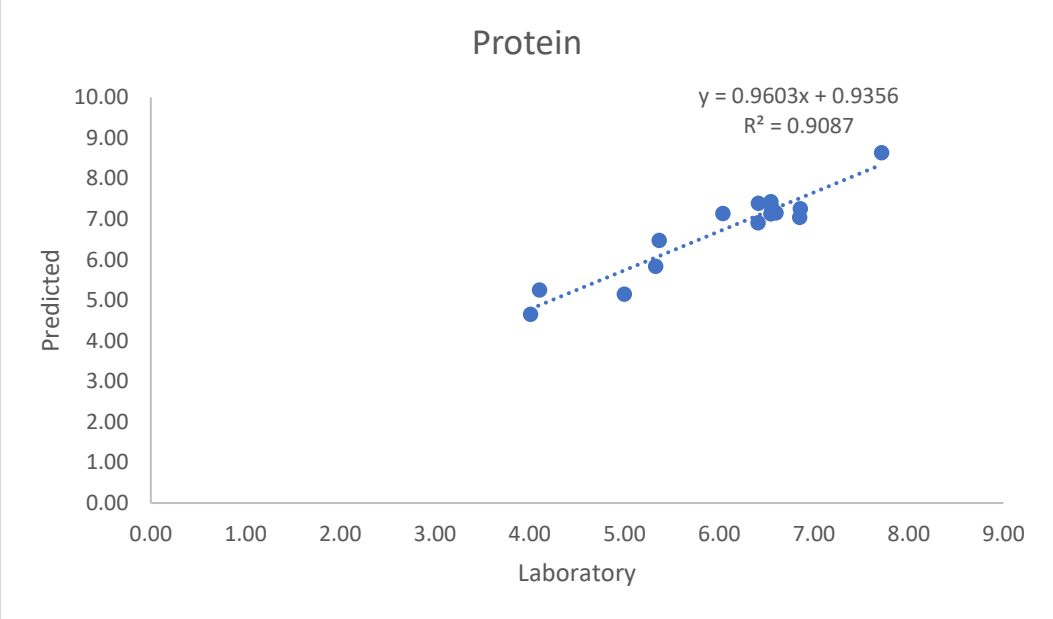

Figure 3: Graph of predicted values vs laboratory values for protein

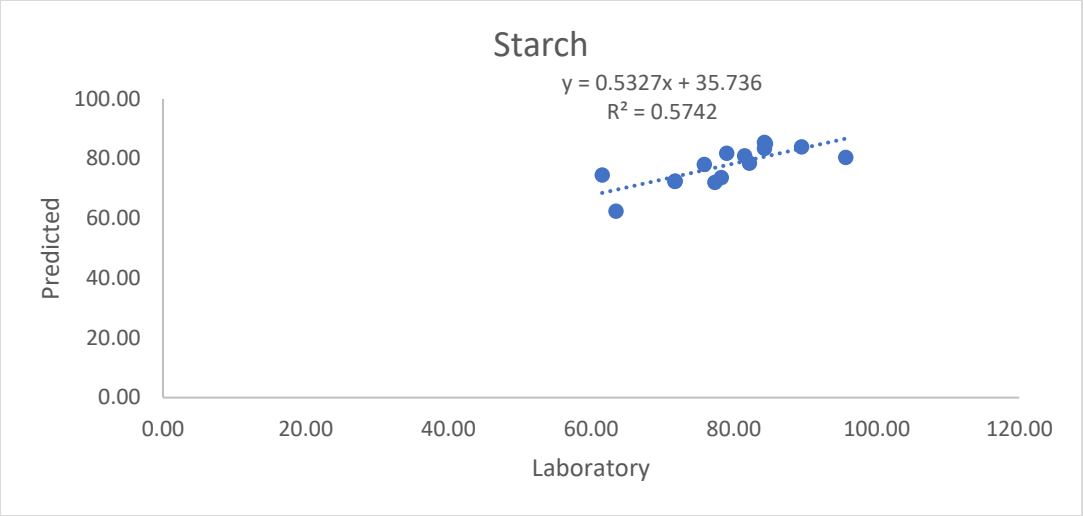

Figure 4: Graph of predicted values vs laboratory values for starch content

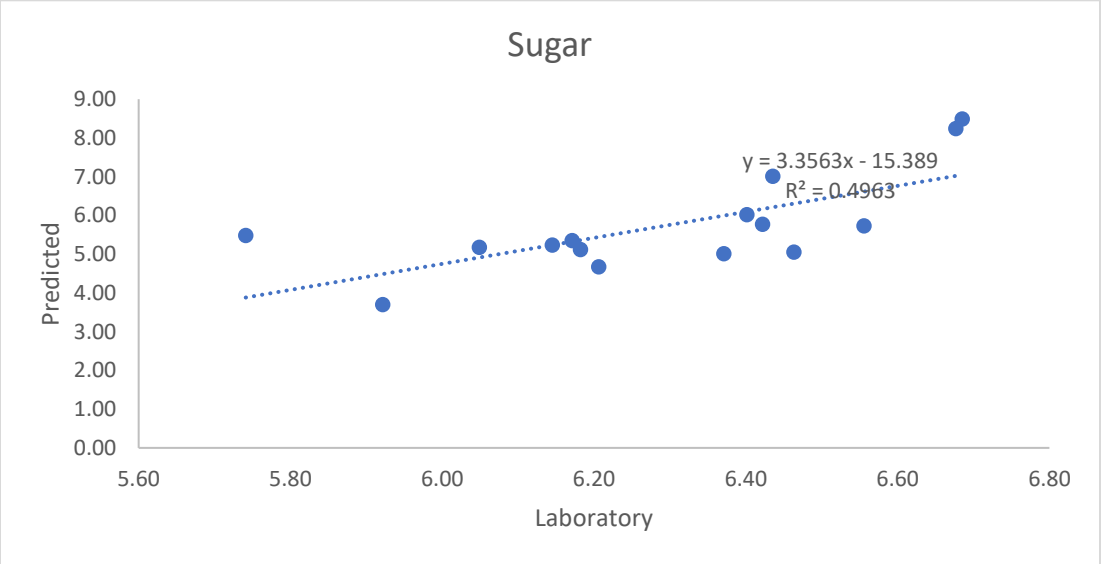

Figure 5: Graph of predicted values vs laboratory values for sugar content

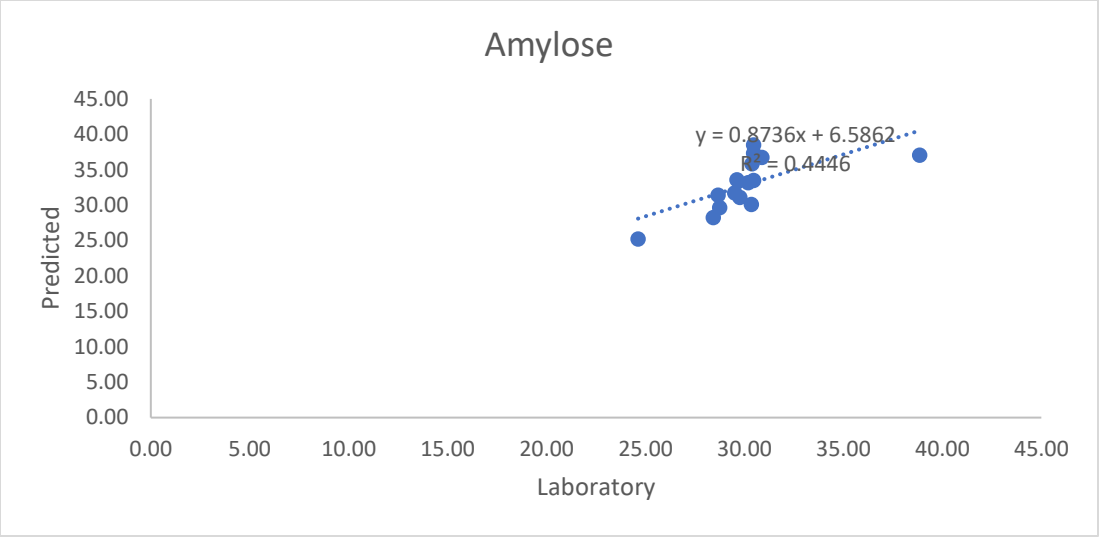

Figure 6: Graph of predicted values vs laboratory values for amylose content

Conclusion:

Proximate as well as antinutrient composition are important parameters to be determined for evaluating the quality of yam and many other crops. NIRS offers a high throughput and less expensive alternative to the elaborate wet chemical analysis in the laboratory for the analyses of yam flour. The equations developed from the study have shown good prediction performance for parameters such as tannin content, moisture content, ash, protein and crude fibre, hence they can be used for the estimation of these parameters in yam flour samples. The models for parameters which have low RSQ values such as fat and phytate can be improved upon because of their low SEC values; 0.14 and 0.33 respectively. It can be concluded therefore that near infrared spectrometers could be used for accurate prediction of tannin content, moisture content, ash, protein and crude fibre of dried yam flour samples.