Quantifying Dry Matter and Titratable Acidity of Fufu-Mash Using Portable Near-Infrared Spectrometer

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Abstract— A traditional fermented paste from cassava is called Fufu. Lactic acid bacteria's activity during the fermentation of this product causes unique biochemical changes in cassava starch. The proper retting of the soaked cassava roots results from effective fermentation, which is influenced by the dry matter of the cassava roots and lactic acids. Measuring these traits in the lab takes a lot of time and money. Therefore, this study aimed to use near-infrared reflectance spectroscopy (NIRS) to develop calibration models for dry matter and total titratable acidity. Eighty cassava genotypes were processed into Fufu mash. The Fufu mash samples were divided into two groups of seventy Fufu samples each for calibration, and ten Fufu samples for validation to create the calibration model. In addition, the lab analysis of the Fufu mash samples' dry matter content and total titratable acidity produced the reference data. The results showed good coefficients of determination in calibrations (R2cal) and cross-validation (R2cv) for dry matter and total titratable acidity, respectively, of 0.83, 0.65 and 0.93, and 0.57. Thus, enhancing the NIRS calibration models used in this study may be possible and using them as a quick screening tool for cassava breeding initiatives aimed at producing Fufu.

Keywords— Cassava, Dry Matter content, NIRS, Total Titratable Acidity, *Fufu* Mash.

I. INTRODUCTION

More than 500 million people worldwide use cassava (Manihot esculenta Crantz), a tropical root crop, due to its prominence and importance as a calorie source [1]. According to [2] cassava has emerged as the primary root crop in Nigeria, contributing significantly to the country's food supply, income, and employment opportunities. Nigeria primarily produces gari, fufu, and lafun from cassava. The majority of farming households in Nigeria produce and use these goods for subsistence usage [3]. People from the southern, eastern, and western parts of Nigeria, as well as those from other parts of West Africa, produce fufu as a product of fermentation [4].

Lactic acid bacteria aid in the breakdown of starch in the cassava root during fufu processing as fermentation advances [5]. Researchers have discovered that the presence of lactic acid accelerates the retting process of cassava roots. Additionally, one of the biochemical factors involved in cassava retting is dry matter content [6]. The cost, effort, and time needed to determine the dry matter content (DM) and total titratable acidity (TTA) in the laboratory are high. Also, well-equipped laboratories are required. The lack of a quick and reliable method to test the chemical properties of raw materials in the lab has made it challenging to quickly breed cassava varieties that meet the needs of end users. Visible near-infrared spectroscopy (vis/NIRS) provides a quick, nondestructive substitute for the traditional study of several constituents [7]. Visible near-infrared spectroscopy is environmentally safe, economically effective, and requires little to no sample preparation [8]. This technique to quickly assess the physiochemical quality and pasting abilities of sweet potato starch [8]. However, no published studies have utilized high-throughput technology such as the handheld vis/NIRS to measure the dry matter contents and total titratable acidity of fufu mash. Therefore, the objectives of this work are to determine the dry matter content and total titratable acidity of wet fufu paste (wet fufu mash). We will achieve this by using a portable ASD Quality Spec vis/NIRS device as a high-throughput method of analysing fufu paste in the laboratory. Additionally, we will develop a calibration equation to predict the dry matter content (DM) and total titratable acidity (TTA) of the paste.

A. Raw Materials and Method for Fufu Processing

We harvested 80 fresh cassava roots from the CET (Clonal Evaluation Trial) of the NextGen Cassava Breeding Program at the National Root Crops Research Institute's Umudike location and processed them into wet fufu mashes. We randomly selected three cassava roots, comprising big, medium, and small sizes, to represent each of the cassava clones. We peeled, washed, submerged them in about 2 liters of water, covered them, and allowed them to ferment for 72 hours. 72 hours is the optimal duration for achieving adequate fermentation [9]. Following the 72-hour fermentation period, we sieved the now retted and soft roots, then drained or decanted the water to obtain the wet fufu mash (Fig. 1).

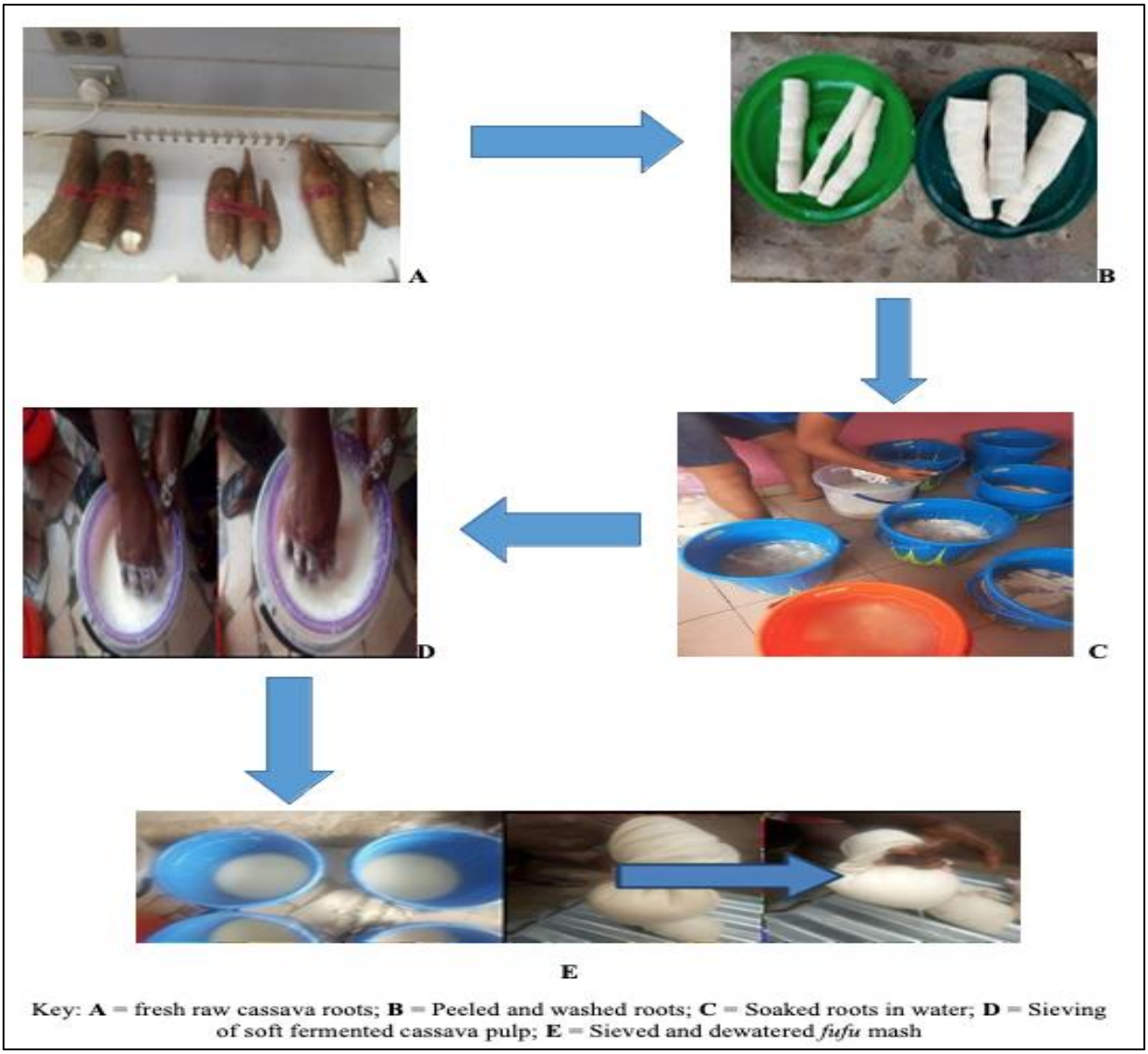

Fig. 1. Workflow for the Processing of Fufu Mash

B. Laboratory Analysis for Reference Result

1) Dry Matter Content Analysis: The dry matter content was calculated as the percentage of dry weight when compared with the fresh weight of the samples after it has been oven dried. The method adopted is as described by[10]. At a steady temperature of 105˚c, 10 g of the homogenized fufu mashes were dried in the oven for 24 hours, and they were done in two replicates. The average dry matter content of the two replications was used for analyses. To calculate the dry matter content of the resulting dried fufu samples, the formula below was used:

$$\% \text{ dry matter} = 100\% - \% \text{ moisture content} \quad (1)$$

2) Determination of Total Titratable Acidity: To determine the lactic acid content, 25 mls gotten from the supernatant liquid of the substrate was titrated against 0.1 m of Sodium hydroxide (NaOH) by adding 3 drops of phenolphthalein indicator. This was done while being shaken vigorously until the formation of a pink colouration. 1 ml of 0.1M NaOH used in the titration is equal to 90.08mg of the lactic acid.

$$\text{Total titratable acidity of lactic acid} = \frac{ml\ of\ NaOH\ x\ N\ NaOH\ x\ M.E}{Volume\ of\ sample} \quad (2)$$

Where: ml NaOH = Volume of NaOH, N NaOH = Molarity of NaOH, M.E represents the Equivalent factor, which is equal to 90.08 mg.

3) Vis/nirs Spectral Measurements of the Wet Fufu Mash: We used method [11] to capture the spectra of the fufu mashes. We filled the ASD quartz sampling cups with 8–10 g of fufu mash, leveled them, and covered them with the white cap cover. We filled three cups with the fufu samples and collected three spectra from each cup. We averaged the nine resulting spectra

to efficiently determine the variation within the particle sizes and the sample-holding device. We collected the spectra of the wet fufu mashes in triplicate, within the wavelength range of 350–2500 nanometres (nm). We registered the absorbance values of log (I/R), obtained at 0.5 nm intervals for each of the fufu samples, using a portable vis/NIRS instrument (Qualityspec Trek: S-10016) from ASD, PANalytical Products, Malvern Panalytical B.V., Lelyweg 1 (7602 EA), PO Box 13, Almelo 7600 AA, Netherlands. We used the Win-ISI 4.9 software from Infrasoft International and FOSS, Hillerod, Denmark to analyze the data and perform the statistical analyses. The near-infrared spectra are often affected by noise from the instrument, the size of the sample particles, and other environmental factors. Because of this, the spectra need to be pre-processed to get rid of any bias that may have formed before the model is made.

4) NIRS Model Calibrations: We used WIN ISI software (version 4.0) for the preprocessing of all the spectra and the calibration of the model. With the use of suitable preprocessing methods, removing noises that emanate from the background, which serve as interferences, becomes critical, as this will help foster the accuracy of the model calibration. The spectral data coll we applied adequate preprocessing methods to the spectral data collected in this work to correct any potential light scattering interference and to strengthen and increase the signal-to-noise ratio. After experimenting with various mathematical treatments, including 1, 10, 10, 1, 0, 0, 1, 1, and 2, 5, 5, 1, along with the standard normal variate and detrend (SNVD), we found that treatment 2, 5, 5, 1 yielded the best results, which in turn helped to optimise the calibration equation. h treatment was assessed adequately so as to get the best treatment that provides a model that is reliable. The first and second numbers in the treatment give the derivative and the gap, while the remaining last two numbers represent the smoothings. The distances of NH and GH, called 'Mahalabonis,' which is the distance between each of the spectra from the total mean spectrum of the whole sample, were used to eliminate the outliers. The work of the 'NH' is to perform a calculation of how each sample is very close to all other samples in the whole population, while the 'GH' performs the action of identifying if the calibration model can predict correctly the values of other unknown samples, thereby giving room for removing the unwanted spectra from the calibration population. The outliers are usually removed from the population using the standard residuals, which have a cut-off of GH > 2.5 and NH < 0.6. We established the calibration from the collected spectra using the corrected first derivative of SNVD, which we calculated based on four data points. We then used the Savitzky–Golay polynomial smoothing process to achieve smoothing. We set up the calibration model using a modified partial least square (MPLS) regression algorithm, with a spectra range of 800 to 2,400 nm (Figure 2).We divided the eighty (80) fufu samples into two groups, using their laboratory results as the reference: 70 samples served as the calibration set, and 10 samples served as the validation. The laboratory results, which served as a reference for the traits analysis, exhibit a good correlation with the spectral data of all the fufu samples collected using the vis/NIRS instrument. We developed NIRS equations through regression using modified partial least squares (MPLS) on the first derivative of the spectra's reflectance and transmittance, applying a math treatment of D = 2, G = 5, S1 = 5, S2 = 1, and scatter correction of SNVD (standard normal variance and de-trend) for all the traits analyzed [12].

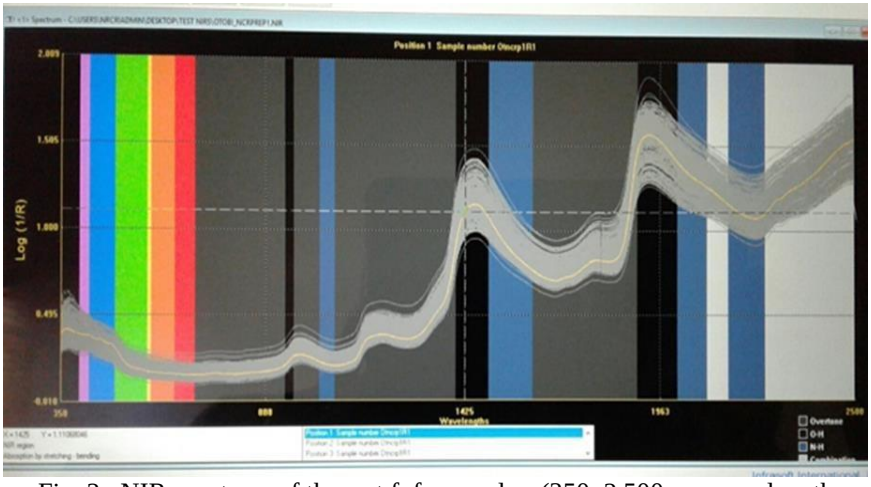

Fig. 2. NIR spectrum of the wet fufu samples. (350–2,500 nm wavelength

5) Model Performance Assessment: We assessed the calibration model performance using the coefficient of calibration determination (R2c), the coefficient of cross-validation determination (R2cv), and the standard error of cross-validation (SECV). Generally, we base optimum models on those with higher R2c values. The R2cal and R2cv values show the performance of the equations when placed under the same conditions [13]. This data set indicates a wide variation with regard to the chemical traits analyzed. The dry matter content of the fufu samples has a standard deviation of 13.22, while the TTA is 3.76. Also, the average TTA of the wet fufu mash samples is equal to 4.93%.

II. Results

Table 1. Summary statistics of the DM and acidity of the *fufu* mash samples

Trait	Min	Max	Mean	SD
DM	40.42	48.42	42.48	3.24
TTA	13.12	38.10	4.93	3.74

Table 2. Calibration statistics for the wet *fufu* mash samples

Trait	No	SEC	R^2c	SECV	R^2cv	Outliers
DM	70	10.42	0.83	3.46	0.93	1
TTA	70	13.13	0.65	2.43	0.57	0

IV. Discussion

The statistical data for the dry matter content and total titratable acidity of the analysed wet fufu mash samples are as presented in Table 1 above. The data showed that the dry matter content ranged from 40.42 to 48.42%, while the total titratable acidity ranged from 13.12 to 38.10%, with mean values of 42.48% and 4.93%. We can compare the mean dry matter content (13.63%) observed in this work with the values obtained by Awoyale et al., 2021b [14]. Dry matter, defined as the total particles remaining after draining or removing water from a food material, serves as an indicator of the readily available nutrients in that particular food material. For any cassava to become desirable, it must have adequate dry matter content. In fact, dry matter highlights the quality characteristics that a good cassava possess high yields, resistance to malfunction and pests, and high fresh and dry yields in the root satisfy consumer needs [15]. Achieving a good dry matter content is the easiest way to increase the storage life and market appeal of fufu flour [16].

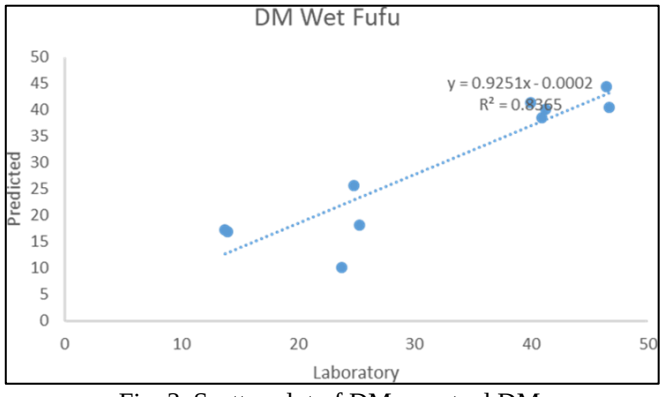

Fig. 3. Scatter plot of DM vs actual DM

The NIRS calibration equations that were made showed that the dry matter content and total titratable acid content had good calibration determination coefficients (R2c) of 0.83 and 0.65, but lower cross-validation determination coefficients (R2cv) of 0.93 and 0.57, respectively Table 2. Often, we categorize determination coefficients (R2) of 0.50 to aid in identifying sample concentrations; we use values from 0.60 to 0.82 for screening and quantification. In the majority of samples, R2 values between 0.83 and 0.90 are crucial, while R2 values between 0.92 and 0.96 are widely used in quality assurance analysis, and R2 values above 0.98 are used in all applications [17]. This data shows minimal variations in R2c. The R2cv indicates that there was homogeneity in the calibrations.

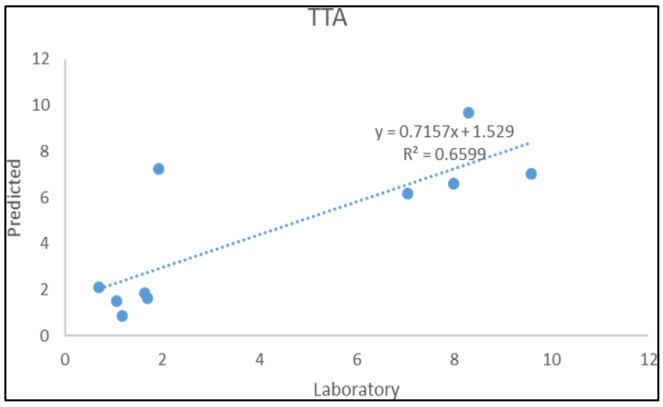

Fig. 4. Scatter plot of TTA vs actual TTA

V. Conclusion

The results of this work indicate that VIS/NIRS has the potential to quickly predict the dry matter content (DM) and

total titratable acidity (TTA) of fufu mash samples. This method of analysis will help remove greatly the tediousness, time wasting, as well as the high cost associated with wet chemical analysis in the laboratory. With this method of analysis will significantly alleviate the tediousness, time wasting, and high cost associated with wet chemical analysis in the laboratory. remove the tasking nature of sampling and preparation of the samples, which usually takes longer time and days, especially during the laboratory activities.

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