*Laboratory Standard Operating Procedure*

# Determination of Dry Matter Content of Wet Fufu Mash using Handheld NIRS

## High-Throughput Phenotyping Protocols (HTPP), WP3

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Ethics: The activities, which led to the production of this document, were assessed and approved by the CIRAD Ethics Committee (H2020 ethics self-assessment procedure). When relevant, samples were prepared according to good hygiene and manufacturing practices. When external participants were involved in an activity, they were priorly informed about the objective of the activity and explained that their participation was entirely voluntary, that they could stop the interview at any point and that their responses would be anonymous and securely stored by the research team for research purposes. Written consent (signature) was systematically sought from sensory panelists and from consumers participating in activities.

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# RTBfoods

***WP3: High-Throughput Phenotyping Protocols (HTPP)***

## SOP: Determination of Dry Matter Content of Wet Fufu Mash using Handheld NIRS

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# ABSTRACT

‘Fufu’, a fermented wet paste food product from cassava is a good source of dietary energy and is one of the widely consumed food in Nigeria and other West African countries. A rapid and accurate method for determining dry matter (DM) content of fufu mash will be valuable for further work to be done on fufu. Portable/handheld NIRS are flexible tools for fast and unbiased analyses of constituents with minimal preparation. This SOP discusses the procedural steps involved in the determining the dry matter (DM) content of wet fufu mash samples with the use of portable/handheld NIRS device. Three different quartz cups were filled with fufu mashes (of about 8-10g) and covered with a white cover material. They were placed against the window of the portable/handheld NIRS device and spectra captured on them. To ensure repeatability of the procedure, nine (9) spectra were captured on one of the samples (TME 419) which gave an overall root mean square of 38452. Random comparison of the sample’s spectra gave 1, 2 = 32182, 1, 3 = 21581, 1, 4 = 35437, 1, 5 = 19887, 1, 6 = 35645, 1, 7 = 14899, 1, 8 = 32498, and 1, 9 = 20162. The values from the compared spectra were seen to be lower than the overall root mean square (RMS) value, which showed good repeatability.

**Keywords: fufu, NIRS, DM, Root mean square**

# 1 SCOPE AND APPLICATION

This SOP discusses the method involved in NIRS analysis to determine the DM content of wet fufu mash samples using a portable handheld NIRS device. The quantity (g) taken are done to be representative of the whole lot of the samples to be analysed.

# 2 REFERENCES

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# 3 DEFINITIONS

DM : Dry matter

PYT : Preliminary Yield Trial

# 4 PRINCIPLE

Near infrared spectroscopy (NIRS) is one of the most important analytical techniques based on the vibrational properties of atoms in molecules (Stuart, BH. 2004). The Near infrared (NIR) spectroscopy is based on the absorption of electromagnetic (EM) radiation at wavelengths in the range 350 - 2,500 nm. The light interacts with the sample and the detector measures its transmittance and absorbance. Transmittance refers to the amount of light that passes completely through the sample and strikes the detector. Absorbance is a measurement of light that is absorbed by the sample. The absorptions measured by NIR spectroscopy correspond mostly to overtones and combinations of vibrational modes involving C–H, O–H, and N–H chemical bonds (Osborne *et al,* 1993). Recording the electromagnetic radiation absorbed from those molecular bonds in the NIR wavelengths produces spectra which are unique to a sample acting as a "fingerprint". The collected spectrum includes data related to the chemical and physical properties of organic molecules in the sample and, therefore, important information on sample composition (dos Santos *et al,* 2013).

# 5 APPARATUS

Portable vis/nirs device (qualityspec trek: s-10016) from ASD, PANalytical Products, Malvern Panalytical B.V, Lelyweg 1 (7602 EA), PO Box 13, Almelo 7600 AA, Netherlands

# 6 PROCEDURE

1. Fill 3 different quartz cups with the fufu sample (of about 8 – 10g). The three cups are chosen to serve as replicates for the samples during spectra collection.
2. Cover the already filled sample cups with the white cover (came alongside the portable NIRS device)

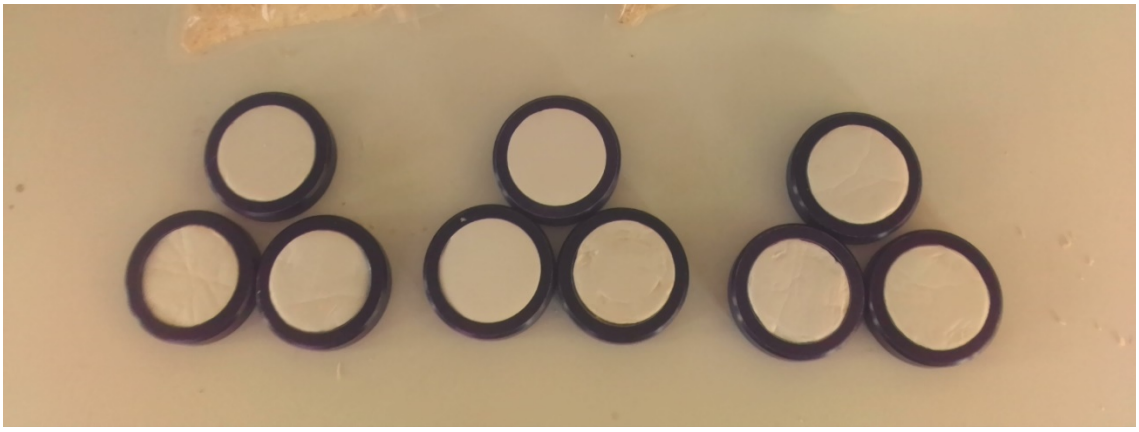

*Figure 1* ***Filled sampling quartz cups***

3. Place the sampling cups against the window of the portable NIRS device and scan. Prior to scanning the samples, spectra of the white reference disc is always taken. This helps check the accuracy of the spectrometer

*Figure 2* ***Spectra collection on fufu mash***

## 6.1 NIRS measurement

Spectral repeatability evaluation

Nine spectra were captured on the fufu mashes samples (3 quartz cups captured 3 times on each cup) for each clone. The quantity to be analysed was homogenised and a portion was taken so as to serve as a representative of the whole lot of the samples to be analysed.

- Numbers of fufu samples : 30

- Spectral range & acquisition mode: Absorbance (350 – 2500 nm)
- Spectrometer: QualitySpec Trek: S- 10016

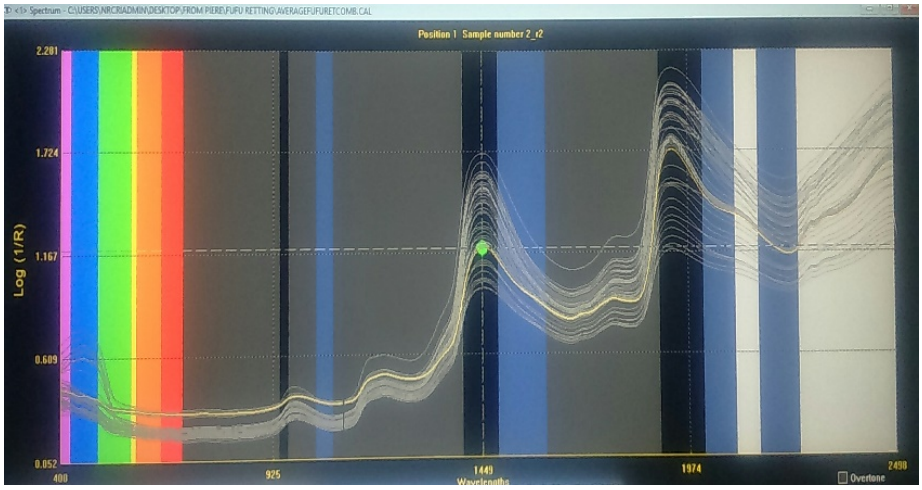


*Figure 3:* ***A typical average visible to near infrared (350–2500 nm)*** **Spectrum illustrating peaks of wet fufu sample**

## 6.2 Repetition

Repeatability refers to the variation that occurs when repeated measurements are made of the same sample under absolutely identical conditions say, same operator–same setup–same units–same environmental conditions etc. it is a way of measuring precision.

*Table showing the spectral repeatability of nine spectra from TME 419*

| Spectra reference | Comparison couples | $RMS_i$ (µabs) |
|---|---|---|
| TME419_1 | 1,2 | 32182 |
| TME419_2 | 1,3 | 21581 |
| TME419_3 | 1,4 | 35437 |
| TME419_4 | 1,5 | 19887 |
| TME419_5 | 1,6 | 35645 |
| TME419_6 | 1,7 | 14899 |
| TME419_7 | 1,8 | 32498 |
| TME419_8 | 1,9 | 20162 |
| TME419 | Average RMS (9 replicates | **38452** |

The table above shows the repeatability of the nine spectra collected on a TME 419 sample. The overall root mean square of the nine replicates was seen to be 38452 uabs. To further show if the spectra is repeatable, random root mean square sampling was done among the nine spectra. The random sampling was done in pairs between sample 1 and 2, 3, 4, 5,6,7,8 and finally sample 9. This is measured by determining the mean, square and sum of squares of the spectra.

From the result in the table, the values were seen to be lower than the overall root mean square value of the nine spectra put together. This shows that the spectra has good repeatability.

According to this result, the decision was made to do 3 replicates per sample

# 7 CRITICAL POINTS OR NOTE ON THE PROCEDURE

1. Ensure the scanned sub-sample is the right representative of the lot to be evaluated.
2. The samples should be well homogenized (in wet form or in dry flour) prior to filling of the cups to get a good absorbance during spectra capture.
3. The size reduction of the dried sample to be analysed should be to a very fine flour (should be milled properly).
4. Prior to spectra capturing, it should be ensured that the cups are well filled with the dry or wet sample with no air spaces in the filling to get homogenised spectra.
5. Ensure that the quartz sampling cups are well placed against the window of the NIRS device (for both the mobile handheld and stationary NIRS devices) so as to get good spectra.
6. For the wet fufu samples, the water content should be about 50% to 70%. This allows for better penetration of the light rays into the sample thereby getting the good wavelength of the constituents of the sample.
7. In the event of deep-freezing the wet fufu samples as a way of storage, it should be ensured that the samples defrost to not less than 50% moisture. This makes the adhesive forces in the sample to be loosened thereby creating room for better penetration of the light rays.

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