Measurement of Oil Content in Dried Palm Mesocarp

Application Note 18

Summary

- Up to 250 times faster than wet chemistry methods*
- No hazardous solvents required; no hazardous waste produced
- Easiest most reliable technique available; suitable for unskilled operators – simple, intuitive visual software
- Simple linear calibration; no chemometrics
- Insensitive to sample colour, particle size and origin

**Not including sample conditioning and drying time.*

Application

Oil palm is grown extensively around the world, chiefly in Indonesia, Malaysia and parts of South America. Crude Palm Oil (CPO) is extracted from the flesh of the oil palm fruit (known as the mesocarp) by mechanical pressing, and is then refined and used for cooking, as a food ingredient, and increasingly as a feedstock for production of biodiesel.

Breeders of oil palm are constantly striving to improve the yields they achieve from their plantations, and one of the ways to do this is to improve the genetic quality of the palm fruits so that they produce more oil. In order to assess and quantify the yield of new strains of oil palm, an accurate and reproducible way of measuring oil content of the palm mesocarp is needed.

Advantages of NMR

Oil content of palm mesocarp has traditionally been measured using Soxhlet extraction (typically with hexane as a solvent), but this is time consuming (typically 16 hours per extraction), has poor reproducibility, and requires the use of hazardous chemicals. By contrast, the Oxford Instruments **MQC+** NMR analyser offers a method that takes only minutes per sample, uses no solvents or other chemicals, and is much more reproducible than the traditional extraction method.

Method

NMR works by causing hydrogen nuclei in liquids to resonate and emit a radio frequency signal. This signal is detected by the instrument, and is directly proportional to the number of hydrogen nuclei in the sample. In the case of palm oil measurement, the NMR signal comes from the hydrogen nuclei in the liquid oil in the palm mesocarp, and is directly proportional to the amount of oil present in the sample.

Calibration

By calibrating the NMR signal against a suitable reference, or against samples of known oil content, we can get a quantitative measurement of oil content. Calibration of the **MQC+** analyser can be performed in one of two ways - either by measuring a series of samples with oil values known from Soxhlet extraction, or by measuring a sample of pure CPO as a 100% oil data point. Whichever method is used, the calibration can be viewed and edited in the **MQC+'s** MultiQuant calibration software and then transferred onto stable, artificial Calibration Maintenance Standards (CMSs) supplied with the instrument. The **MQC+** can, if necessary, be quickly restandardised in future by using these CMSs, removing the need to analyse more mesocarp samples or rely on a CPO sample that may have deteriorated.

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Sample Size

Palm mesocarp is by its nature, inhomogeneous, so there can be significant differences in oil content between different portions of a sample. For this reason it is important to measure as large a sample as possible in order to optimise the measurement statistics. The Oxford Instruments **MQC+** uses 26mm diameter sample tubes, which can hold approximately 5g of dried mesocarp, which is sufficient to ensure good correlation against Soxhlet measurements.

Measurement

The method for measuring oil in palm mesocarp with the Oxford Instruments **MQC+** is as follows:

1. Remove the mesocarp from the fruit then dry overnight in an oven to determine the moisture content (this step is also required for the traditional Soxhlet extraction method).
2. Homogenise the dried mesocarp to reduce sample-to-sample variation.
3. Weigh the sample in a 26mm NMR sample tube then condition in a dry block heater for at least 15 minutes to ensure all oil in the sample is liquid.
4. Measure the sample in the **MQC+** analyser for 16 seconds.
5. Read the oil content from the **MQC+**.

Results

A set of 10 mesocarp samples were measured by Soxhlet extraction. Seven of these samples were used for calibration and another three were reserved as calibration test, or validation, samples. In addition to the mesocarp samples, samples of pure CPO and an empty sample tube were also measured to represent 100% and 0% oil respectively. The results are shown in Figure 1 below, where red dots represent calibration points and green squares represent validation samples.

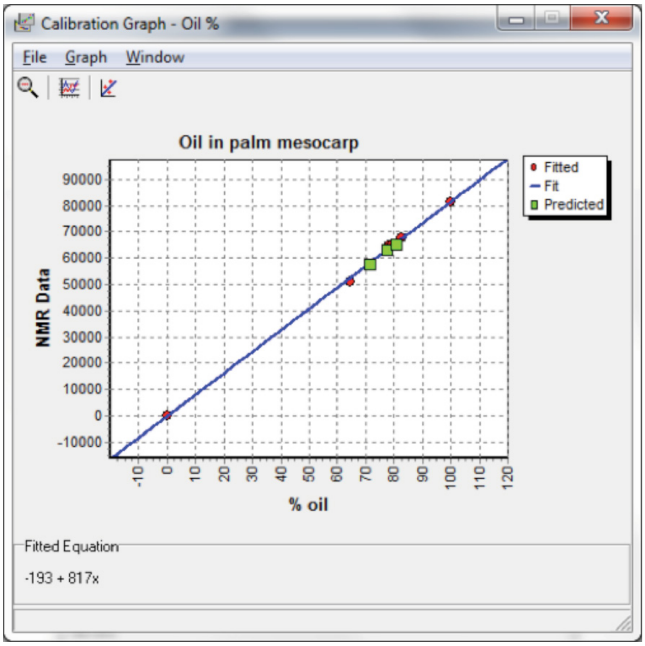

Figure 1: *NMR calibration for oil in palm mesocarp tissue.*

The results for the validation samples were as shown in Table 1 below.

Validation sample No.	Reference Soxhlet value (% oil)	NMR measured value (% oil)	Difference (%)
1	71.5	70.72	-0.78
2	77.3	77.39	0.09
3	81	80.19	-0.81
Standard deviation of prediction samples			0.51

Table 1 - *Comparison of the Soxhlet vs. predicted NMR values for % oil in palm mesocarp*

Errors and repeatability

The dominant error in measurement of oil in palm mesocarp comes from the natural variability of the sample. Measurements on different portions of the same sample can differ by several percentage points, as shown in Table 2. This error feeds through into the NMR calibration because we cannot be sure what is the "correct" Soxhlet value to use as the reference. The same table however also shows that NMR measurements on the same portion of sample are highly repeatable and precise, giving confidence in accurate measurements once a good calibration has been generated. As a guideline, a "good" calibration line should go through 100% and 0%.

Sample 3619	Repeat 1	Repeat 2	Repeat 3	Repeat 4	Repeat 5	SD
Portion 1	75.87	75.91	75.90	76.01	75.98	0.05
Portion 2	79.14	79.14	79.29	79.30	79.33	0.08
Difference	3.27	3.32	3.39	3.29	3.35	

Table 2 – *NMR repeatability of portions taken from a single palm mesocarp sample*

Conclusion

The Oxford Instruments **MQC+** NMR analyser offers a fast and precise method for determination of oil in palm mesocarp without any need for chemical solvents. Its benefits are:

- A primary calibration can be produced, quickly and simply, using a single sample of oil which in practice can cover a concentration range of 0 to 100%.
- NMR is very stable over the long term and rarely needs calibration adjustment. If required, this can be done simply using stable Calibration Maintenance Standards which recreate the original calibration carried out during installation.
- NMR is virtually insensitive to sample colour, particle size, or sample origin.
- NMR penetrates through the whole sample and is insensitive to air voids, which means it provides the most accurate measurement of the total amount of oil in a given volume of sample.
- The measurement repeatability is typically better than 0.1% oil.
- The NMR technique is non-destructive so the same sample may be measured several times before being analysed by other techniques.
- Sample measurement time is rapid (16 seconds)*.

Oxford Instruments Ready-to-run Application Package

- **MQC+**23 which can be controlled using its own built-in computer operating Microsoft Windows or via a stand-alone PC
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Oil in Dried Palm Mesocarp' application
- A set of three Calibration Maintenance Standards (CMSs) for calibration maintenance and quality control
- Test/tuning sample
- 26 mm glass tubes
- PTFE stoppers (to seal the tubes)
- Stopper insertion/removal rod
- Installation manual
- Method sheet

In addition you may require:

- A dry block heater and aluminium block with holes for sample conditioning
- A precision (3 decimal place) balance

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