Application Note AN-NIR-044

Multiparameter Quality Control of Palm Oil with NIR Spectroscopy

FFA, iodine value, moisture, DOBI, and carotene content measured

Determination of key quality parameters of palm oil, namely free fatty acids (FFA), iodine value (IV), moisture content, deterioration of bleachability index (DOBI), and carotene require the use of several different analytical methods, which are laborious and can lack in accuracy.

This application note demonstrates that the XDS RapidLiquid Analyzer operating in the visible and near infrared spectral region (Vis-NIR) provides a **cost-efficient and fast solution** for the determination of these quality control parameters in palm oil. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows for the analysis of palm oil in **less than a minute** and **can be used by anyone**.

EXPERIMENTAL EQUIPMENT

Palm oil samples (crude palm oil) were measured in transmission mode with a XDS RapidLiquid Analyzer over the full wavelength range (400–2500 nm). Reproducible spectrum acquisition was achieved using the built-in temperature control (at 60 °C) of the XDS RapidLiquid Analyzer. For convenience, disposable vials with a path length of 8 mm were used, which made cleaning of the sample vessels unnecessary. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

Figure 1. XDS RapidLiquid Analyzer and a palm oil sample present in a 8 mm disposable vial.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
XDS RapidLiquid Analyzer	2.921.1410
Disposable vials, 8 mm diameter, transmission	6.7402.000
Vision Air 2.0 Complete	6.6072.208

RESULTS

The obtained Vis-NIR spectra (Figure 2) were used to create prediction models for quantification of the individual key parameters. The quality of the prediction models was evaluated using correlation diagrams, which display the correlation between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

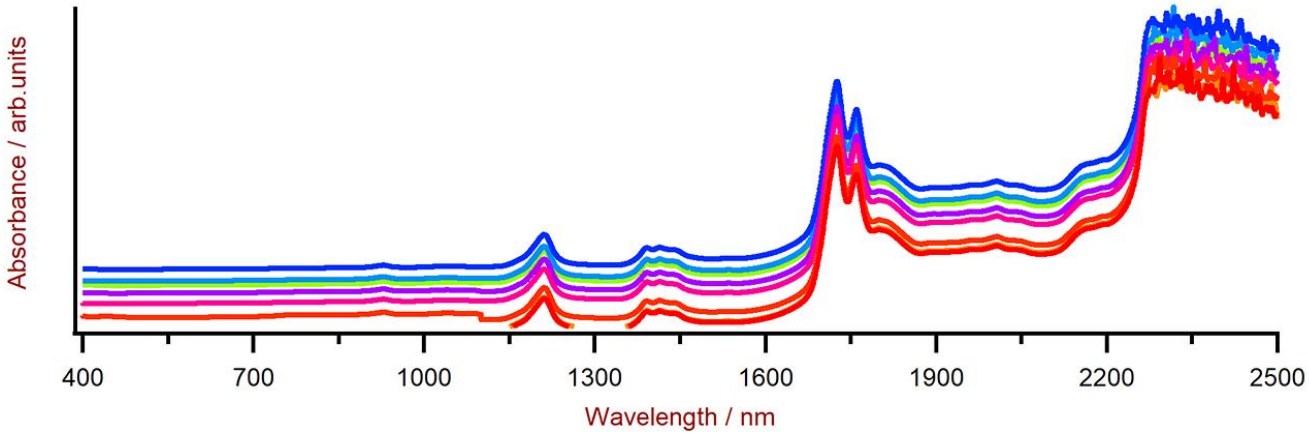

Figure 2. Selection of palm oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.

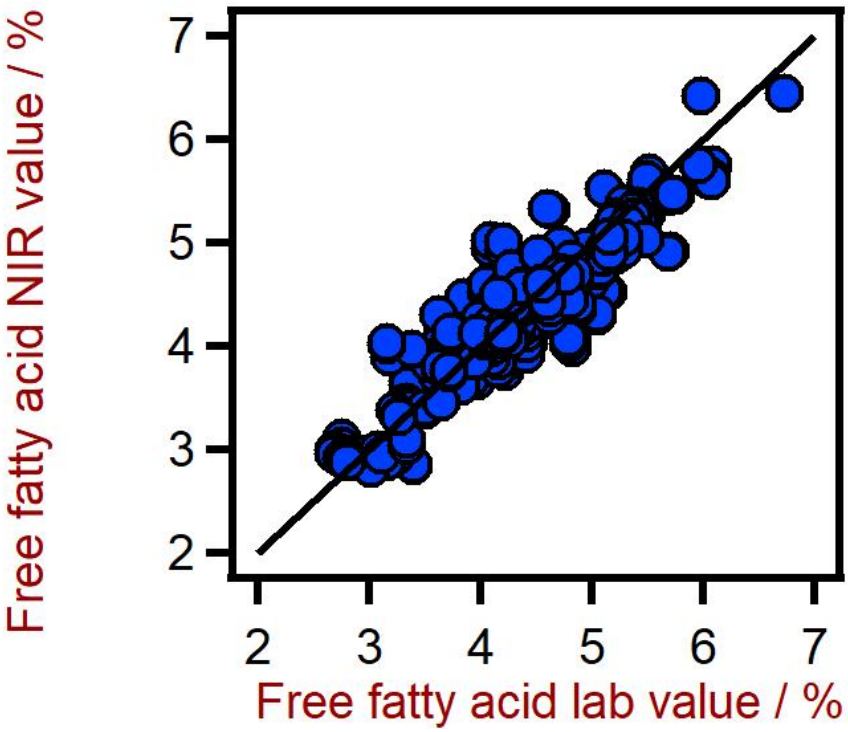

Figure 3. Correlation diagram for the prediction of the result free fatty acid in palm oil using a XDS RapidLiquid Analyzer. The free fatty acid lab value was evaluated using titration.

Table 2. Figures of merit for the prediction of the free fatty acids in palm oil using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.835
Standard error of calibration	0.266%
Standard error of cross-validation	0.270%

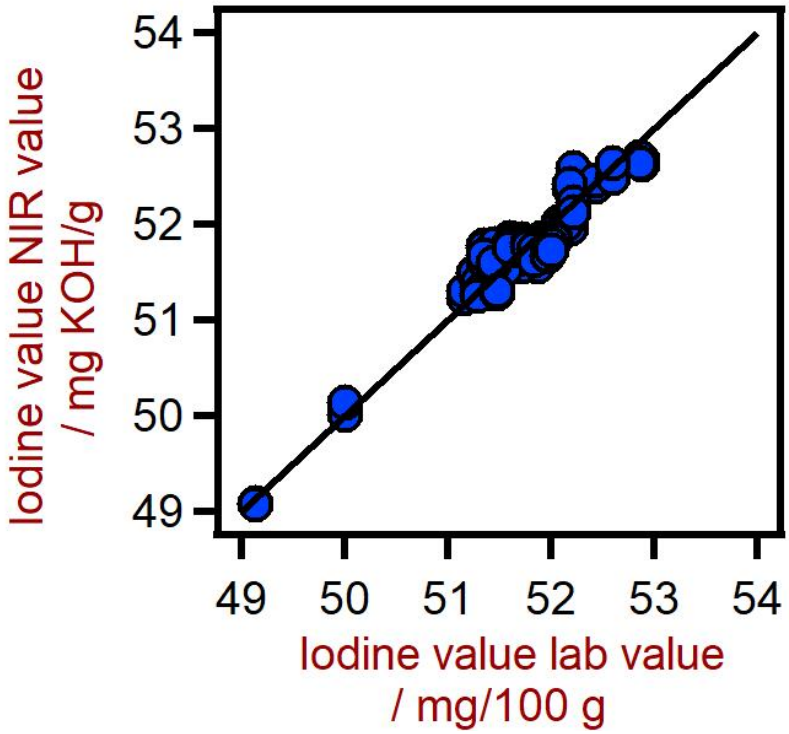

Figure 4. Correlation diagram for the prediction of the iodine value (IV) in palm oil using a XDS RapidLiquid Analyzer. The iodine lab value was evaluated using titration.

Table 3. Figures of merit for the prediction of the iodine value in palm oil using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.911
Standard error of calibration	0.184 mg/100 g
Standard error of cross-validation	0.201 mg/100 g

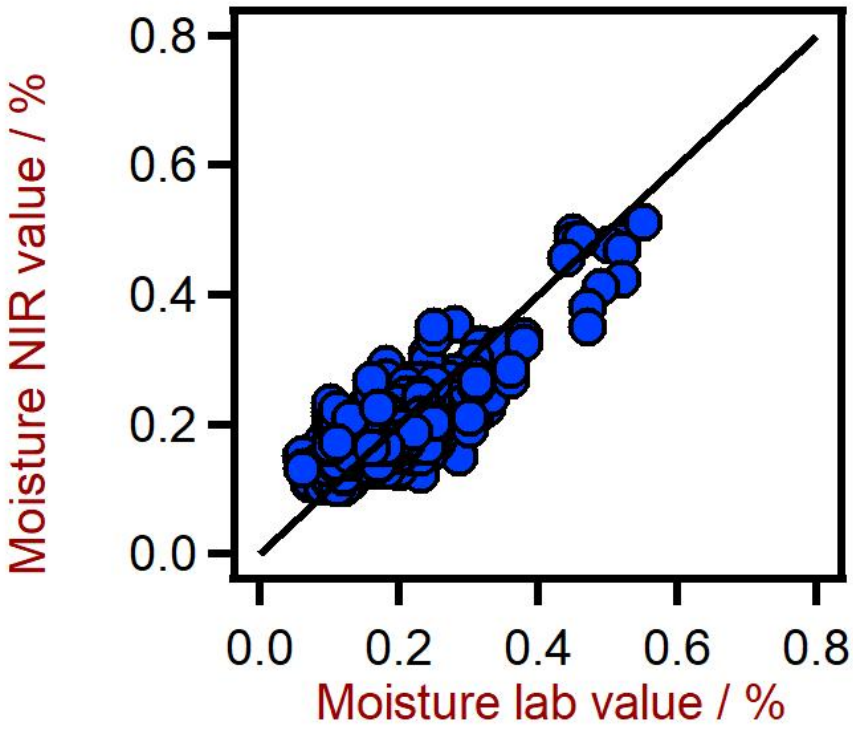

Figure 5. Correlation diagram for the prediction of the moisture content in palm oil using a XDS RapidLiquid Analyzer. The moisture lab value was evaluated using Karl Fischer (KF) titration.

Table 4. Figures of merit for the prediction of the moisture content in palm oil using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.638
Standard error of calibration	0.046%
Standard error of cross-validation	0.047%

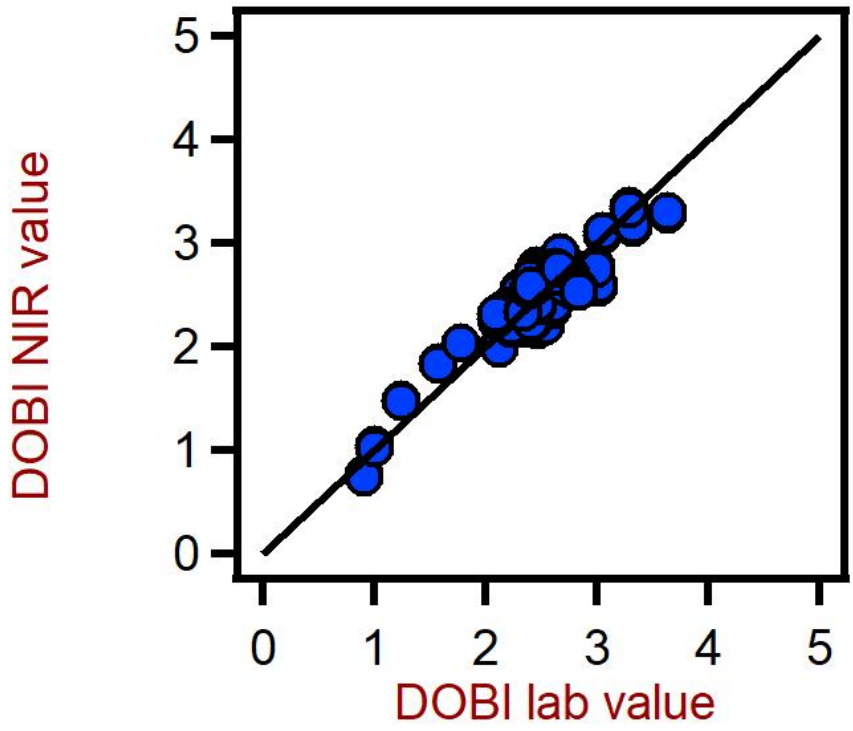

Figure 6. Correlation diagram for the prediction of the deterioration of bleachability index (DOBI) in palm oil using a XDS RapidLiquid Analyzer. The DOBI lab value was evaluated using photometry.

Table 5. Figures of merit for the prediction of the deterioration of bleachability index (DOBI) in palm oil using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.842
Standard error of calibration	0.17
Standard error of cross-validation	0.19

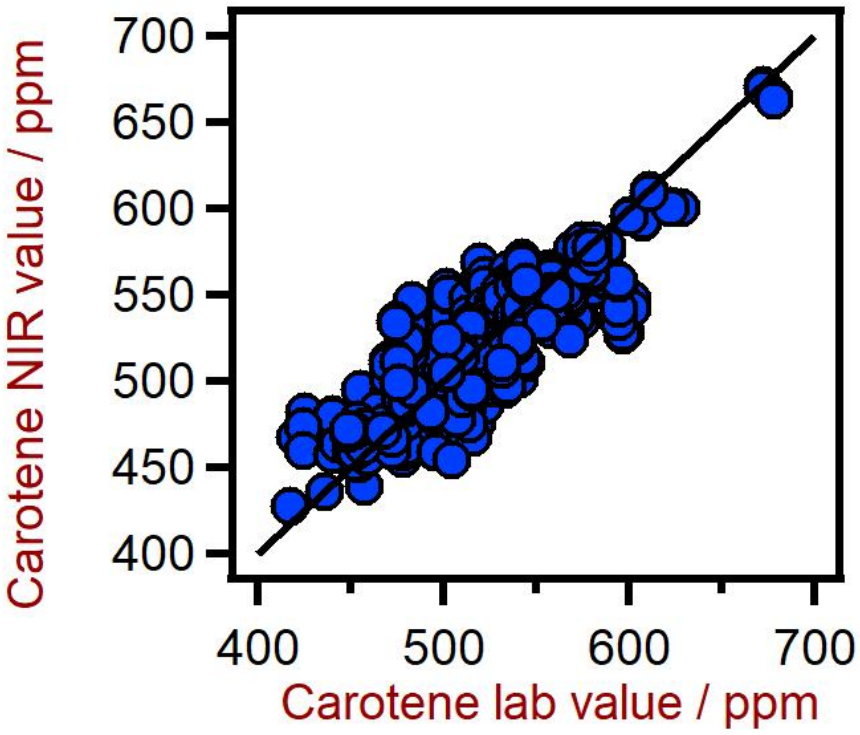

Figure 7. Correlation diagram for the prediction of the carotene content in palm oil using a XDS RapidLiquid Analyzer. The carotene lab value was evaluated using photometry.

Table 6. Figures of merit for the prediction of the carotene content in palm oil using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.677
Standard error of calibration	22.9 ppm
Standard error of cross-validation	23.4 ppm

CONCLUSION

This application note demonstrates the feasibility of NIR spectroscopy for the analysis of the FFA content, iodine value, moisture content, DOBI, and carotene content in palm oil. In comparison to wet chemical methods, running costs are significantly lower when using NIR spectroscopy (Table 7 and Figure 8).

Table 7. Comparison of running costs for the determination of the hydroxyl number with titration and NIR spectroscopy.

	Lab method	NIR method
Number of analyses per day	10	10
Cost of operator per hour	$25	$25
Costs of consumables and chemicals (FFA, IV, moisture, DOBI, carotene)	$9	$1
Time spent per analyses (FFA, IV, moisture, DOBI, carotene)	22 min	1 min
Total running costs per year	$42,900	$2,063

Figure 8. Comparison of the cumulative costs over three years for the determination of key quality parameters in palm oil with titration/photometry and NIR spectroscopy.

CONTACT

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NIRS XDS RapidLiquid Analyzer

Rapid, precise analyses of liquids and suspensions of all types.

The NIRS XDS RapidLiquid Analyzer enables rapid, precise analyses of liquid formulations and substances. Precise measurement results at the push of a button make the NIRS XDS RapidLiquid Analyzer an equally reliable and simple solution for quality monitoring in laboratories and processes. The samples are transferred to quartz cuvettes designed for multiple use or disposable glass vials; a tempered sample compartment ensures reproducible analysis conditions and thus accurate measurement results.

Vision Air 2.0 Complete

Vision Air - Universal spectroscopy software.

Vision Air Complete is a modern and simple-to-operate software solution for use in a regulated environment.

Overview of the advantages of Vision Air:

- Individual software applications with adapted user interfaces ensure intuitive and simple operation
- Simple creation and maintenance of operating procedures
- SQL database for secure and simple data management

The Vision Air Complete version (66072208) includes all applications for quality assurance using Vis-NIR spectroscopy:

- Application for instrument and data management
- Application for method development
- Application for routine analysis

Additional Vision Air Complete solutions:

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)