APPLICATION NOTE

Near-Infrared Spectroscopy

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Quality Control of Olives by Near-Infrared Spectroscopy and AssureID Software

Introduction

Olive oil is an increasingly popular food product worldwide, with global production exceeding 3.0 million tons in 2011 and showing steady annual growth. Despite these huge volumes, however, margins are relatively small in olive oil production so quick and easy analysis of oil quality is vital to maintain process efficiency. Rapid, reliable analysis can contribute to process and quality improvements in numerous ways. For example,

- Assessment of raw olive acceptability. If the olives have been collected from the ground rather than fresh from the tree, they may be of poor quality with high acidity and hence lower value.
- Measurement of water and oil content. These parameters determine the price of the olives, with those having a greater oil content commanding a higher price.
- Process optimization. After extracting the oil, the remaining pulp or by-product (called alperujo in Spain) should have only minimal oil content, typically around 2% or less. If the oil exceeds this level, a problem with the process is indicated.

This note describes how a PerkinElmer Frontier™ FT-NIR spectrometer and AssureID software have been used by an olive oil producer in Spain to improve productivity by implementing the above analyses within their routine production.

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Materials and Methods

A PerkinElmer Frontier FT-NIR spectrometer equipped with an upwards-facing reflectance accessory and sample spinner (NIRA) was used for all measurements.

Olive samples were milled to a paste and placed in a glass petri dish before analysis. Spectra were collected between 10000 and 4000 cm^{-1} at 16 cm^{-1} resolution, with an accumulation time of 30 seconds per sample.

The olive samples were also analyzed for oil and water content following the customer's established laboratory procedures.

Some of the measured spectra are shown in Figure 1. Typically for NIR spectra, the absorption features are broad and overlapped, although several prominent features can be assigned either to water or to organic C–H modes in the oil.

Assuring Olive Quality

SIMCA is a powerful chemometric method for sample classification that builds independent models for each sample class – in this case, fresh and old olives. New samples are tested against both models, and identified as belonging (or not) to one of the material classes. Compared to traditional methods of identification such as spectral correlation, SIMCA has a much greater ability to distinguish between relatively similar materials, even in the presence of natural variation – provided this is captured in the training set data used to build the models.

AssureID software was designed from the ground up to streamline the process of building SIMCA models, and breaks the procedure down into a series of straightforward, logical steps:

1. Define materials and acquire spectra of known references.
2. Optionally, configure algorithm parameters and spectral pre-processing such as baseline correction: the default settings are tailored to the instrument and sample type and in most cases will produce good results without modification.
3. Calibrate the method. The software automatically builds the models and determines the acceptance thresholds.
4. Review the classification results (for example, see Figure 2). Any issues with the data or performance of the method will be flagged by the troubleshooting engine, allowing corrective action to be taken.
5. The validated method is then deployed as a workflow within the dedicated Analyzer module of AssureID, allowing routine use of the method.

Quantitative Modeling of Oil and Water Content

The oil and water contents of the olives are key parameters for quality, and both contribute to the NIR spectrum. The complex nature of NIR spectra often makes it impossible to develop quantitative models based on the absorbance at a single wavelength. However, multivariate (chemometric) methods such as partial least squares regression (PLS) still function in the presence of overlapping bands, and can allow models to be built.

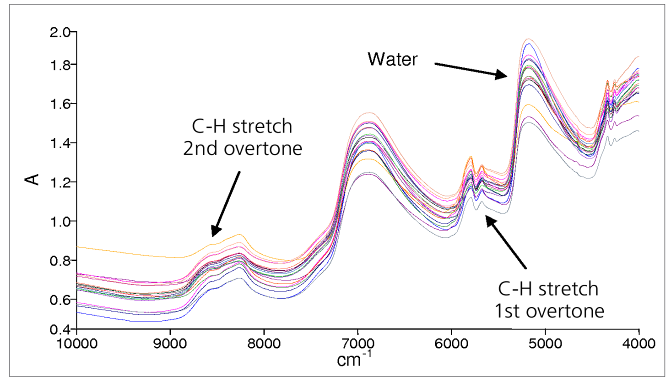

Figure 1. NIR spectra of some of the olive samples.

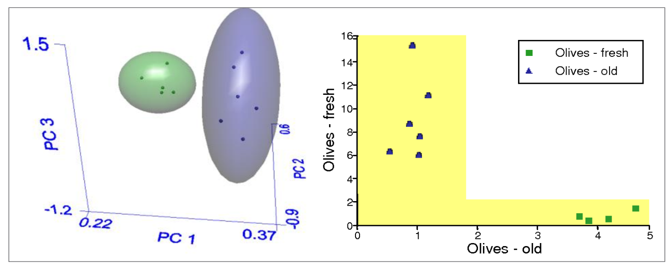

Figure 2. Overview PCA (left) and Cooman's (right) plots for the models to discriminate old and fresh olives. Each axis represents the residual distance against one model. A clear separation of points in the top-left and bottom-right corners, as seen here, indicates that the model is comfortably distinguishing the two types of olive.

The olive spectra and properties determined by chemical analysis were loaded into PerkinElmer Quant+ software. One third of the data were designated as a validation set to verify the performance of the model. The spectra were pre-processed with first-derivative baseline correction.

The calibration and validation results are summarized in Table 1 and Figure 3. The models use a modest number of latent variables and show good linearity and precision over the range of available samples. The standard errors of prediction (SEPs) were 1.5 % and 1.7 % for oil and water, respectively.

Table 1. Summary of results for the PLS modeling of oil and water in olives.

Property	Oil (%)	Water (%)
Range	14–41	34–61
Mean	25	46
Standard deviation	5	6
No. of latent variables	5	3
Validation SEP	1.5	1.7

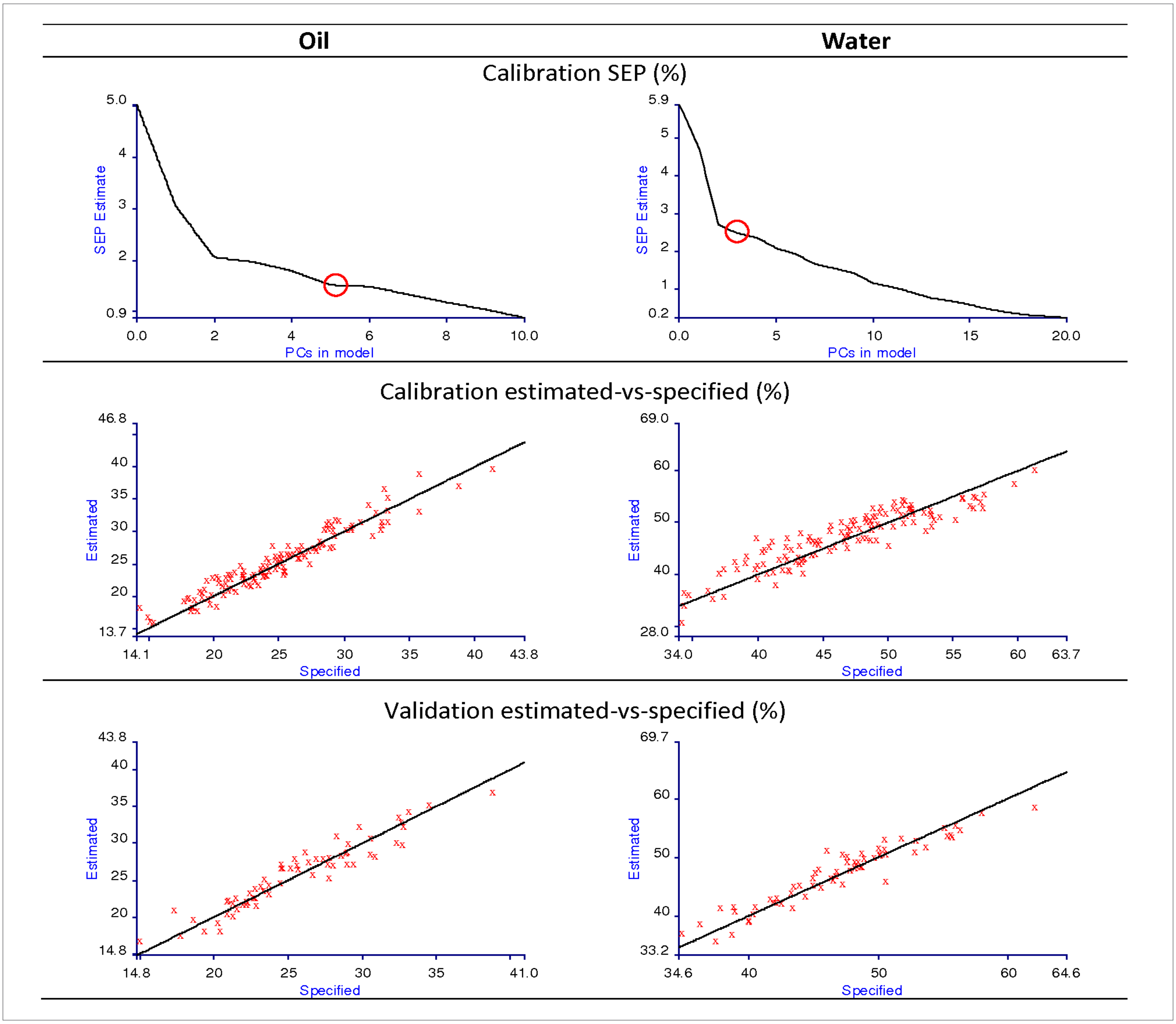

Figure 3. Calibration and validation results for oil (left column) and water (right column).

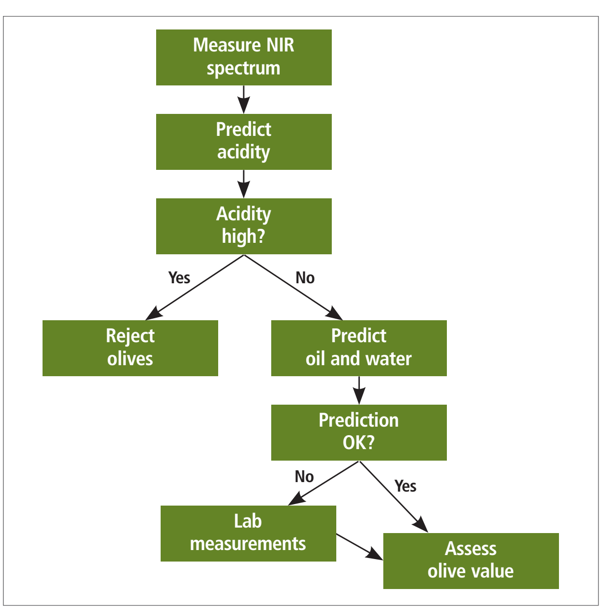

Figure 4. Flow chart for olive analysis by NIR spectroscopy with AssureID.

These quantitative models were also incorporated into the AssureID analysis. After checking the olives for quality, acceptable olives will be further analyzed for oil and water content (as shown in the flowchart in Figure 4).

While AssureID allows sophisticated analyses using both qualitative and quantitative chemometric methods, its design as separate method-building and analysis modules ensures that the end-user is presented with a simple interface, as shown in Figure 5.

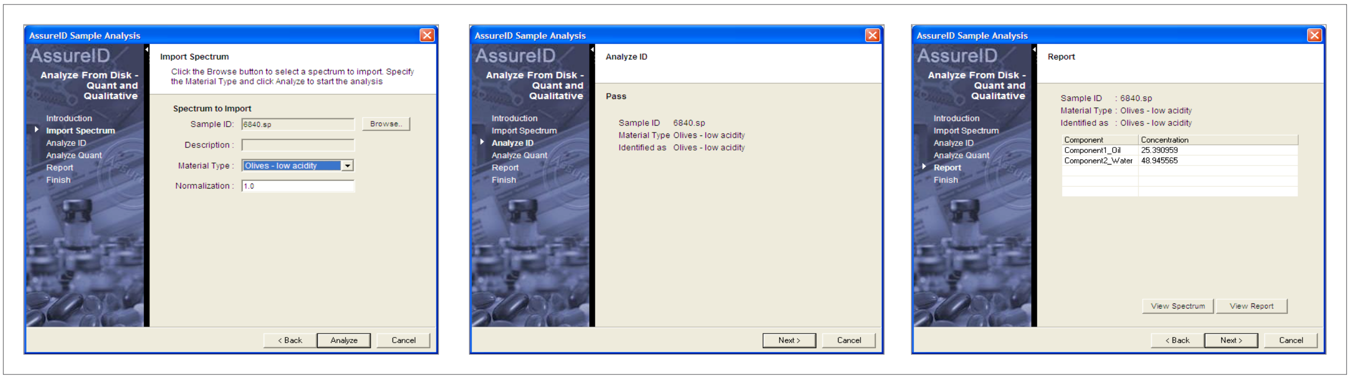

Figure 5. AssureID workflow for olive analysis.

Conclusions

Increasing pressure on food and food ingredient manufacturers to increase efficiency while maintaining product quality has created a need for rapid and precise analysis of materials at all stages of the processing chain. Near-infrared spectroscopy provides rich information about physical and chemical properties of many food materials, and combined with chemometric techniques can provide unequalled speed and precision of analysis. In this note we have shown how the Frontier near-infrared spectrometer from PerkinElmer, in conjunction with AssureID software, is being used to perform three key analyses in olive processing: checking for excess acidity to reject poor-quality olives, measuring the oil and water content to assess olive value, and measuring the oil content in the alperujo by-product to verify extraction efficiency.

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