

FT-NIR Spectroscopy

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Rapid Determination of Solid Fat Content of Specialty Fat Using FT-NIR Technique

Introduction

Differential Scanning Calorimetry (DSC) and Nuclear Magnetic Resonance (NMR) spectroscopy are commonly used to determine the crystallization

behavior of solid fat content (SFC) in fats. These techniques are time consuming and laborious. In the NMR technique, hourly tempering at 60 °C, 0 °C, and 25 °C is essential before the degree of crystallization is measured at 10 °C, 20 °C, 25 °C, 30 °C, and 35 °C.

In the manufacturing of specialty fat, crystallization behavior is extremely important to ensure the quality of the finished product. A crystallization process, to promote the solid phase of the product to be suspended in the liquid phase, is an established way to overcome the sedimentation that occurs during prolonged storage. Homogenous solid suspension with great flow can be achieved when large homogenous crystals are formed, with lesser surface area in the product, which minimizes the possibility of coalescence. As the crystallization process is very fast, a rapid approach to measure crystallization is essential.

The application of chemometric approaches using partial least-squares regression (PLS) is becoming more widespread owing to the availability of digitized spectroscopic data and fast computers. Chemometric modelling approaches are shown to be applicable for the determination of SFC.

In this application study, Fourier Transform Near-Infrared (FT-NIR) was used to determine SFC in palm oil and other edible oil products. FT-NIR spectroscopy with PLS calibration methods was used to quantify SFC at various temperatures: 10 °C, 20 °C, 25 °C, 30 °C, and 35 °C. The predictive abilities of FT-NIR with PLS methods were investigated.

Experimental

Instrument Settings and Calibration Models

A PerkinElmer Spectrum Two N FT-NIR (Figure 1) was used to collect transmission spectra of oil blends of specialty fats in 8 mm glass vials. The spectra were collected with 16 scans from 14700 - 2000 cm^{-1} at a spectral resolution of 8 cm^{-1} . Calibration models were built for each temperature studied (10 °C, 20 °C, 25 °C, 30 °C, and 35 °C) using the PerkinElmer Spectrum Quant software. The spectral range 9000 - 4500 cm^{-1} was used for the analysis, with an Upper Absorbance Limit of 2.5 absorbance units, using first derivative pre-processing with a noise reduction value of 10.

Results

The analysis involves scanning the sample in a heated liquid cell and processing the data, requiring only about one minute to complete the entire analysis. The raw NIR absorbance spectra from the 10 °C calibration standards are shown in Figure 2. The calibration curves in Figure 3 show good correlations between the specified SFC content determined by NMR and the FT-NIR predicted values. Table 1 shows that the NMR values and the FT-NIR prediction results have high variances (R squared) ranging from 81 - 92%. These may be further improved with the addition of more standards added to the model. FT-NIR is shown to be used as a rapid and alternative approach for the determination of SFC to the NMR method.

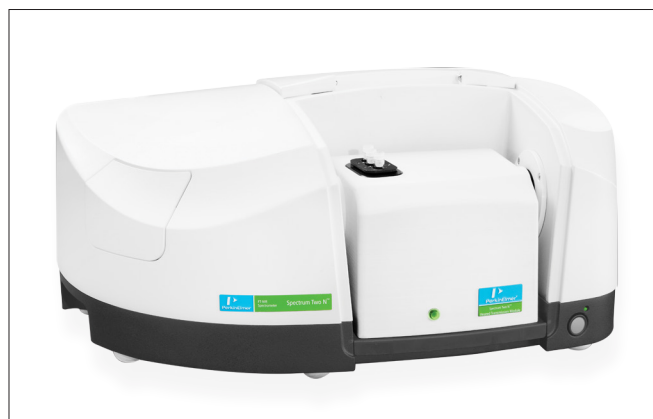


Figure 1. PerkinElmer Spectrum Two N FT-NIR with Heatable Transmission Module.

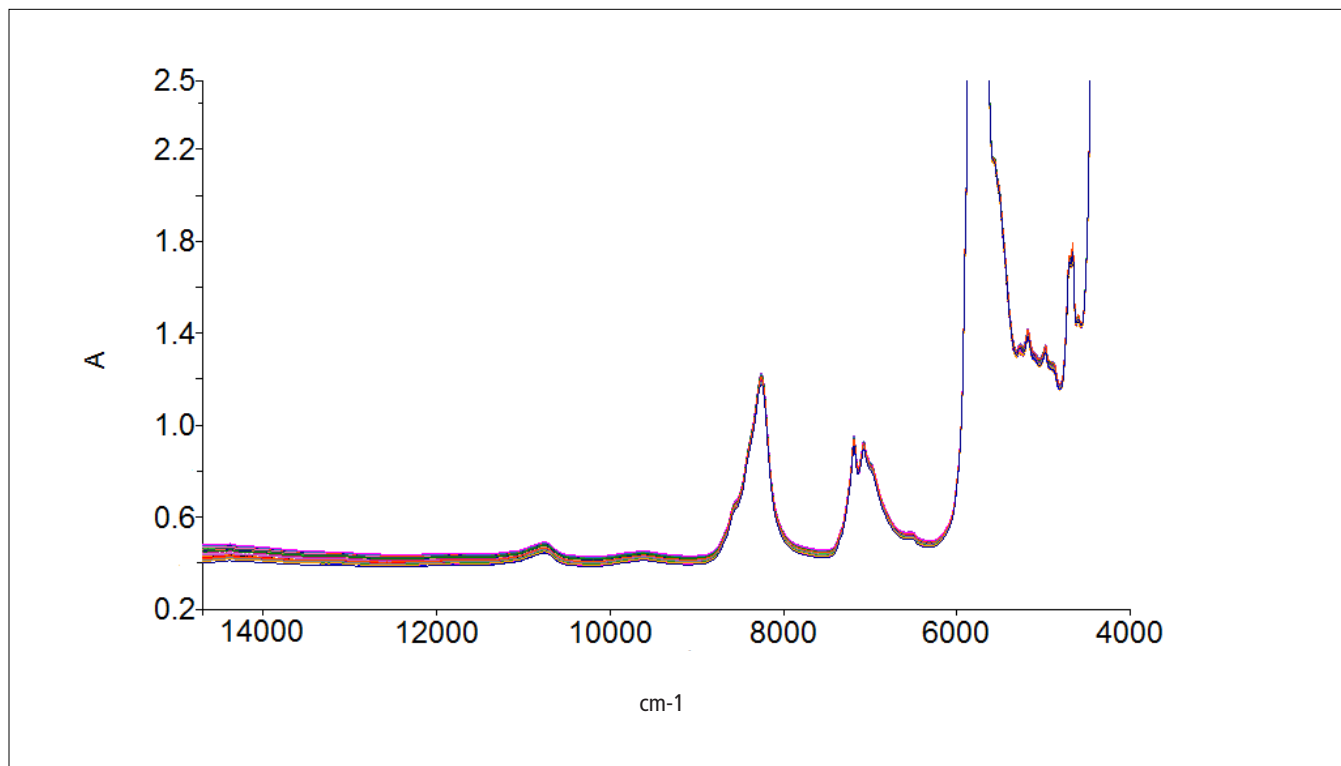


Figure 2. NIR spectra of specialty fat standards.

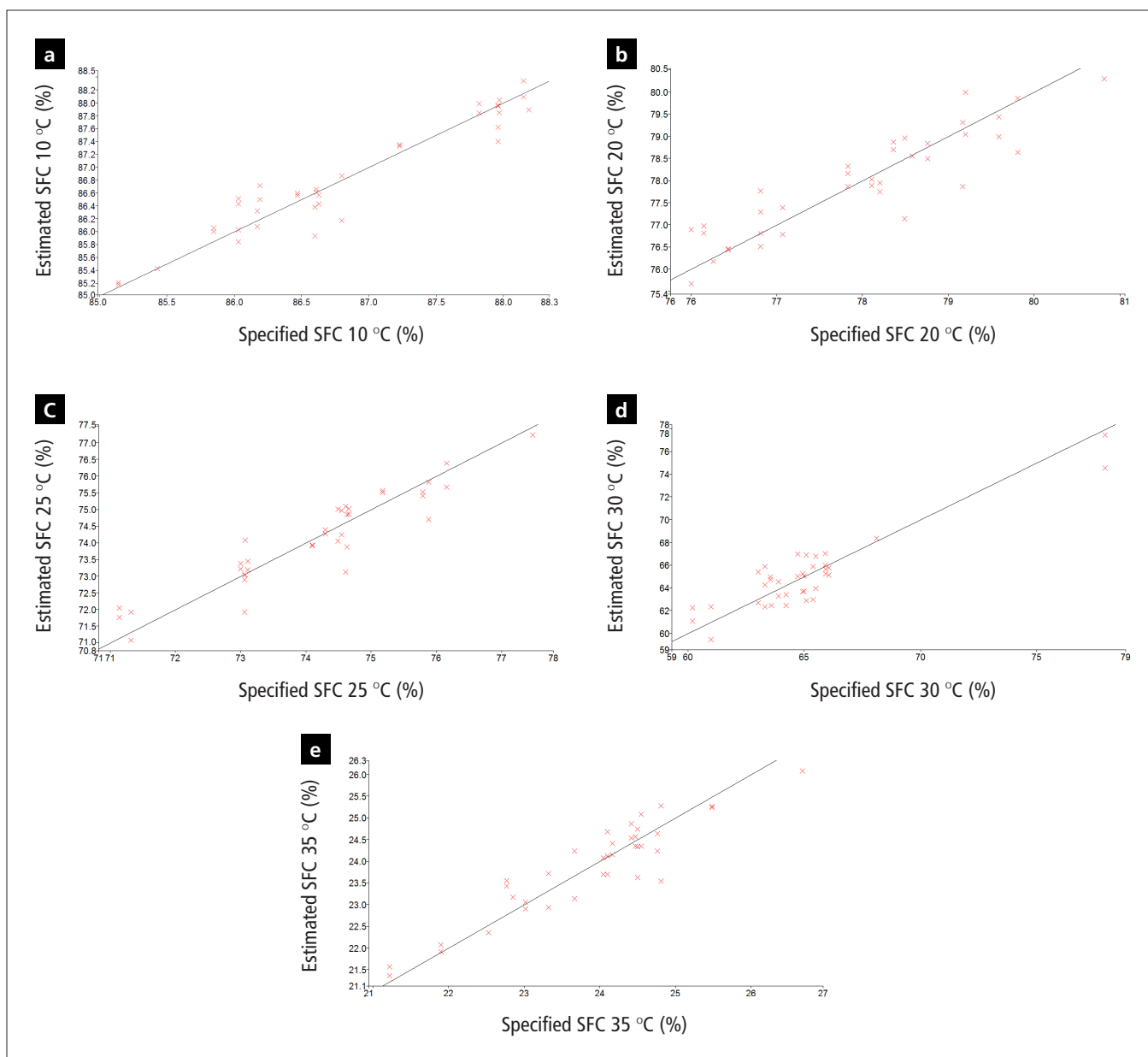


Figure 3. Calibration models for Specialty Fats-Solid Fat Content (SFC) at 10 °C (a), 20 °C (b), 25 °C (c), 30 °C (d) and 35 °C (e).

Table 1. Calibration Summaries for Solid Fat Content of Specialty Fat.

Chemometric Calibration Summary		
Property	Number of Principal Components (PCs)	% Variance (R squared)
10 °C	4	91.55
20 °C	4	80.93
25 °C	5	86.80
30 °C	4	83.82
35 °C	5	85.95

PerkinElmer's Spectrum Two N Oils Analyzer features:

- Preset calibrations or only requiring calibration fine-tuning on site with ready method settings for quick start
- Integrated heater for liquid cell for transmission measurement of the oil samples
- Preheater to warm up samples to required temperature to reduce waiting time
- Absolute Virtual Instrument™ (AVI) (Figure 4) technology for
 - Improved calibration transfer between instruments
 - Accurate wavelength calibrations with all accessories
 - Re-validation or bias correction of calibrated models is usually minimum.

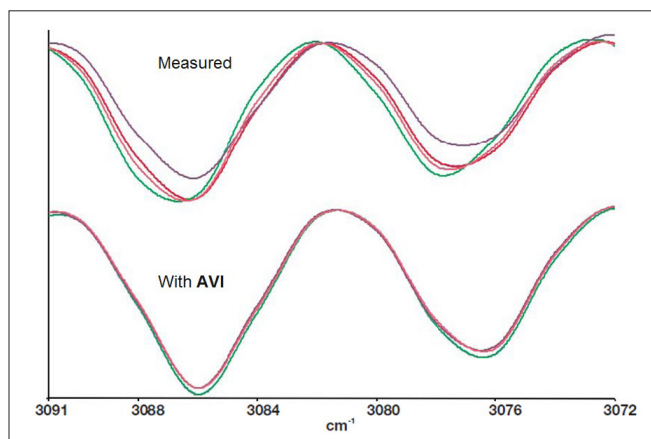


Figure 4. AVI correcting differences between instruments.

Conclusion

The PerkinElmer Spectrum Two N with Heatable Transmission Module is an alternative rapid method to the time-consuming and laborious Nuclear Magnetic Resonance (NMR) reference method for the quantitation of solid fat content (SFC) in specialty fat. The Spectrum Two N FT-NIR can be configured with the temperature-controlled, rugged Heatable Transmission Module suitable for liquid oil transmission analysis. The Absolute Virtual Instrument in Spectrum Two N improves and delivers more consistent performance to FT-IR users in the palm oil industry without requiring routine re-calibration commonly required in traditional dispersive Near-IR technology.

PerkinElmer's Spectrum Two N FT-NIR spectrometer provides a rapid alternative to NMR with high throughput to the specialty fat industry for the determination of solid fat content.