On-line monitoring of crispbread moisture content by short-wave near infrared spectroscopy

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Introduction

The moisture content of rye crispbread is an important quality parameter. Therefore, it is essential to be able to control the baking process by means of an accurate on-line moisture analyser. Typically, the near infrared (NIR) reflectance at 1.94 μm is used for measuring moisture content. However, crispbread is rather thick and due to the high absorption of the 1.94 μm reflectance this would only be capable of measuring the surface moisture. Short-wave near infrared (SWNIR) wavelengths have much lower absorption coefficients, which enable making true transmission measurement and, therefore, measuring the total moisture content of crispbread. Lower absorption coefficients also involve a greater sample volume and there is less need for sample homogenisation. In the SWNIR region, high-sensitivity Si-detectors can be used to gain a high signal-to-noise (SNR) ratio.

A non-invasive SWNIR analyser was implemented to measure the moisture content of crispbread moving on a conveyor. This paper discusses issues concerning the instrumentation and calibration.

Moisture analysis of crispbread

The advantages of using the SWNIR region include the possibility of employing transmission measurements, as mentioned earlier. However, due to overlapping absorption bands, the calibration is more difficult than for the 1.94 μm region. In practice, the use of only a few wavelengths and multiple linear regression (MLR) calibration does not give satisfactory results. It is, therefore, necessary to use a multivariate calibration, such as partial least squares (PLS).

Industrial environments are likely to show temperature fluctuation, which also affects sample temperature. The baking quality of crispbread is also controlled by regulating the oven temperature. Accordingly, the temperature dependency of the calibration will also have to be considered. The effects of temperature variation to the spectrum of water has been widely studied.[1–3] It is known that temperature affects the size of hydrogen-bonded water molecule clusters.[1] Consequently, the wavelength and the intensity of the absorption band of water is strongly dependent on temperature. With PLS calibration, the temperature dependency of water can be compensated by using some additional calibration factors.[4] Water molecules can also build hydrogen bonds with starch. The formation of these hydrogen bonds are dependent on the amount of water present in bread, which causes non-linearity between the absorbance and water content of bread. These non-linearities call for quite a complex calibration, while in PLS this increases the total number of factors.

Experimental

Laboratory experiments were implemented with the Bomem MB-155 Fourier transform infrared (FTIR) spectrometer, using a 1 cm^2 silicon PIN diode detector. Reference moisture was obtained by

comparing the weight of the sample before spectral measurement to the weight after 24 hours of drying in 100°C. Samples were measured in three different temperatures between 25 and 45°C. To prevent drying of the samples during warming and measuring, they were closed in airtight cuvettes. Calibrations were tested by cross-validation.

The results indicate that temperature variation can be controlled with PLS calibration when the calibration set includes the variation in sample temperature. The number of PLS factors had to be increased from five to eight to get similar results for temperature-varied samples to those at room temperature only. The simulations based on FTIR spectra demonstrated that the use of 32 channels from 832 to 1048 nm, each with a half power bandwidth of 13.5 nm, will yield nearly the same calibration accuracy as an FT-IR with a wavelength resolution

Figure 1. The PLS calibration of a 32-channel instrument simulated on FT-IR.

of about 1 nm. In Figure 1, a simulated 32-channel spectrometer calibration is illustrated. In this calibration, the standard error of prediction is about 0.2%.
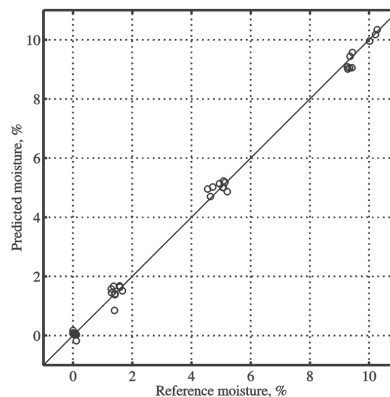
Instrumentation

Light-emitting diode-array spectrometer

A spectrometer design, developed by VTT Electronics, based on a linear light-emitting diode (LED) array light source and a fixed grating monochrom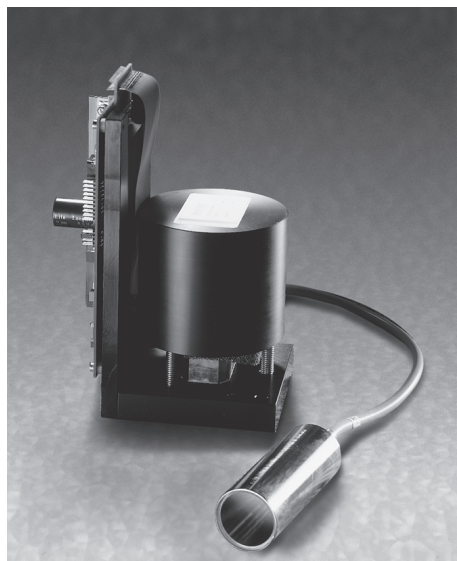ator was used for the measurements. The design employs a powerful, temperature-stabilised 32-element NIR LED array source, built with five different types of surface emitting LEDs, centred at wavelengths 850, 890, 935, 950 and 1020 nm. The assembly procedure included methods for adjusting and permanently fixing the wavelength scale performance; in this case the scale ranged from 832 to 1048 nm. The half-power bandwidth was 13.5 nm and the optical output power, typically, 80 µW for each channel. In the experiments, an SNR level of 10^4, at an absorbance of 3 A ,was achieved, with a 5 s integration time.[5] Spectral scanning was implemented electronically with no moving parts. The scanning speed was limited by the speed of the large area silicon PIN diode detectors. In this application, a 14 ms scanning time was used. A photograph of the LED array spectrometer unit with integrated control electronics is shown in Figure 2.

Figure 2. LED array module with integrated electronics.

On-line analyser implementation

The rye crispbread measurement was implemented as non-invasive, on-line measurement on
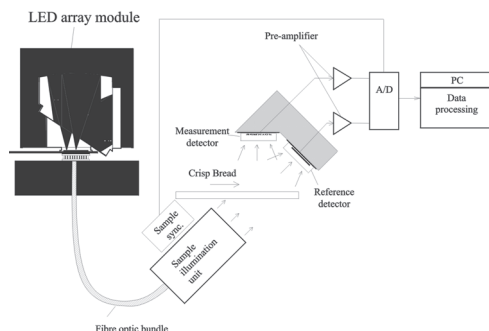
Figure 3. A schematic of the on-line moisture content analyser for crispbread.
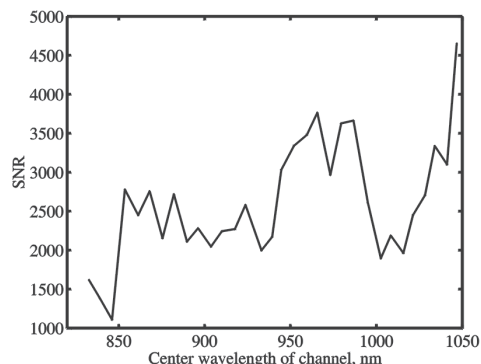

Figure 4. The signal-to-noise ratio for a 2 OD sample without averaging.

moving samples without any sample preparation. The analyser was placed on a conveyor about 30 m after leaving the baking oven. The speed of the sample on the conveyor was 7–10 m min⁻¹. With a 14 ms scanning time, the sample moved about 2 mm during one single scan. Since the diameter of the illuminated spot was about 20 mm, the sample movement did not cause any significant error between the sequentially measured wavelength channels. The thickness of crispbread sample varied from the maximum of 7 to 8 mm to only 0 to 2 mm at the holes. To decrease the effects of thickness fluctuation, the measurement was carried out with a 45° angle between the illumination beam and the detector, as shown in Figure 3. The reference spectrum was measured with a separate detector after the sample had passed the measurement detector. The sample and the reference measurements were timed by detecting the samples with an optical switch just before the measuring beam. The data was collected on a PC with a data acquisition card. The data manipulation software and user interface were programmed with LabView. The spectral data could be saved if additional calibration data was required. Otherwise, the prediction was calculated immediately after measurement. The signal-to-noise ratios of a single spectrum for a 2 OD sample is shown in Figure 4. A low SNR for the first three channels was caused by an 850 nm long pass coloured glass filter, which was used for suppressing interference by ambient light. A photograph of the analyser is shown in Figure 5.

Figure 5. The on-line analyser at a test bench.

Conclusion

An electrically scanned LED array spectrometer can be used for analysing the total moisture content of crispbread on-line. The use of the SWNIR spectral region enables the use of transmission measurement, which is essential to ensuring that total moisture is measured instead of surface moisture. The LED array spectrometer can

be used in diverse applications in on-line or at-line applications, thanks to it having no moving parts and achieving high signal-to-noise ratios for highly absorbing samples. Low power consumption also makes it applicable in portable analysers.

References

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