

# On-line process and quality control by NIR sensor in food industry

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## Introduction

Near infrared (NIR) spectrometry is an ideal technique for the fast chemical analysis and the non destructive quality control of solid and liquid products in industrial conditions. Due to low absorptivity coefficients of the NIR light, direct chemical analysis is possible without sample preparation. Moreover, the NIR radiations at wavelengths between 700 and 1100 nanometers (nm), which are named short-wave near infrared (SWNIR) may be transmitted efficiently across several centimeters thickness of biological matter. The new small spectrophotometers with fibre-optic cable, stationary grating and coupled charge device (CCD) detectors are very simple, robust and sensitive instruments. As they really suit to the automatic analysis of the products by a spectroscopic technique they may be considered as spectrosensors. The application of these sensors in on-line process and quality control has been experimented. The measurement performances of such instruments using the intelligence of calibrated microprocessors will be compared with the requirements of process and quality control of three different food products chosen as models : honey, wheat and apples.

## Experimental

The experiments were implemented with a small fibre-optic spectrophotometer Ocean Optics (Dunedin, Florida) model S2000 equipped with a 1028 pixels Coupled Charge Detector (CCD) and a stationary grating for light measurement at wavelengths between 525 and 1100 nanometers (nm). A 100 Watt tungsten-halogen lamp has been used as light source. A fibre-optic probe collected and guided the transmitted radiations from the sample towards the detector. The light detection was commanded by a personal computer microprocessor according to a home-made software program of data acquisition.

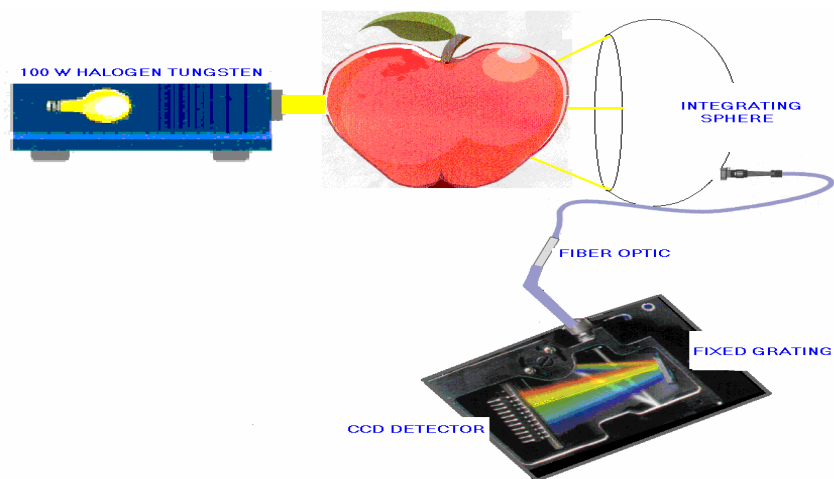
The experimented sample presentation is quite similar for the three types of product. As shown in figure 1, the sample was placed between the light source and the fibre-optic cable terminated by a half integrating sphere in Teflon for the light transmission measurement across apples. The thickness of the transparent glass cell was respectively 3 and 10 centimetres for the transmission measurement across wheat and honey.

In spectrum acquisition, the integration time has been adjusted in order to obtain sufficient and acceptable photoelectric signal of the light intensity  $I_t$  transmitted through the sample and compared with the intensity  $I_0$  crossing a neutral material whose the thickness was adapted to obtain an equivalent scattering effect in the reference and the sample materials. The spectral data were the absorbance  $\log(I_0/I_t)$  values obtained at each wavelength from 525 to 1100 nm.

In data treatment, the calibration equations converting the spectral data into chemical data have been established by the Stepwise Multiple Linear Regression (SMLR) method (Osborne et al., 1993). These calibration equations were computed from the reference chemical data of a set of calibration samples. They were validated after comparison of the predicted and the reference chemical data of validation samples. The correlation coefficient  $R$  and the Standard Error of

Calibration SEC or Validation SEV are two statistical estimators used to evaluate the performances of the calibration.

Among all the food products analyzed in our laboratory three products have been chosen as demonstration models : honey, wheat grains and whole apples. For honey the chemical data were the glucose, fructose and sucrose concentrations, for wheat grains the protein and water content, for apples the mealiness level. The mealiness is an internal defect of texture which depreciates the fruit quality. Fifty to hundred samples of each products have been measured by chemical analysis and spectrophotometry



**Figure 1.** Assembly of light source, sample, fiber-optic probe, fixed grating and CCD detector used in measurement of light transmission across whole apples.

## Results

The spectra of water, protein, fat and sugars in figure 2 should help to understand how it is possible to analyse food products such as wheat grains by computing the  $Y$  values of concentration for each chemical constituent according to the Beer's law and the multilinear calibration equation:  $Y = a_0 + b_1x_1 + b_2x_2 + \dots b_ix_i \dots$  where the  $x_i$  values are the absorbance data of the product at  $i$  different wavelengths. Indeed such a multivariate equation can be a convenient solution of three analytical problems encountered in spectrophotometry of complex biological products : 1. spectral interference, 2. matrix effects, 3. light scattering .

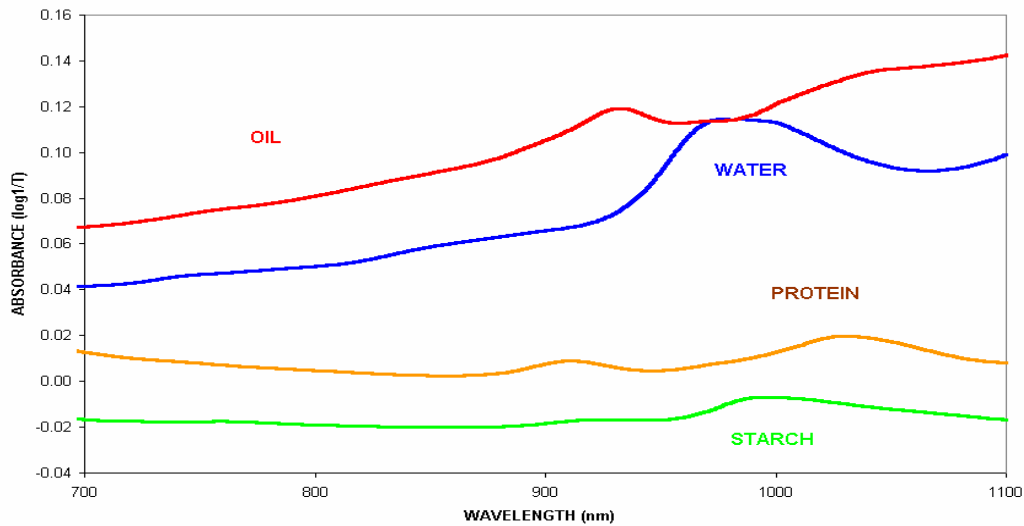


Figure 2. NIR spectra of the main constituents of wheat grains.

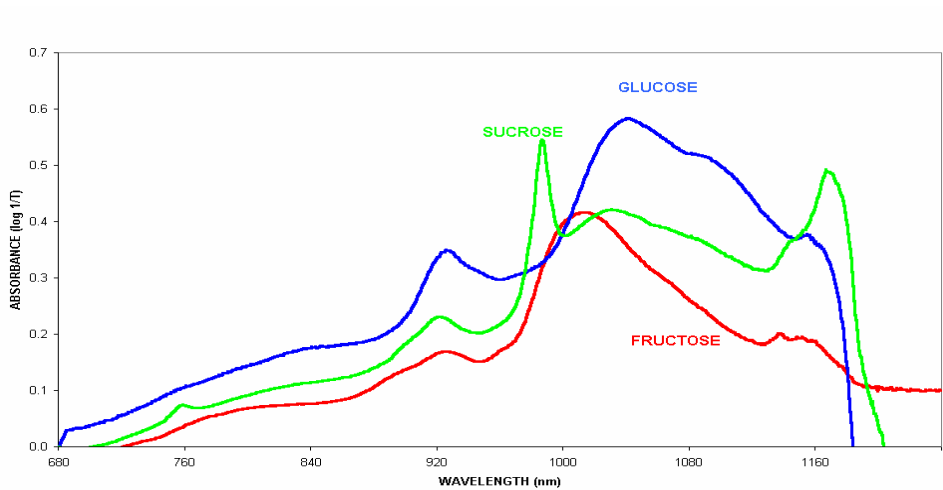
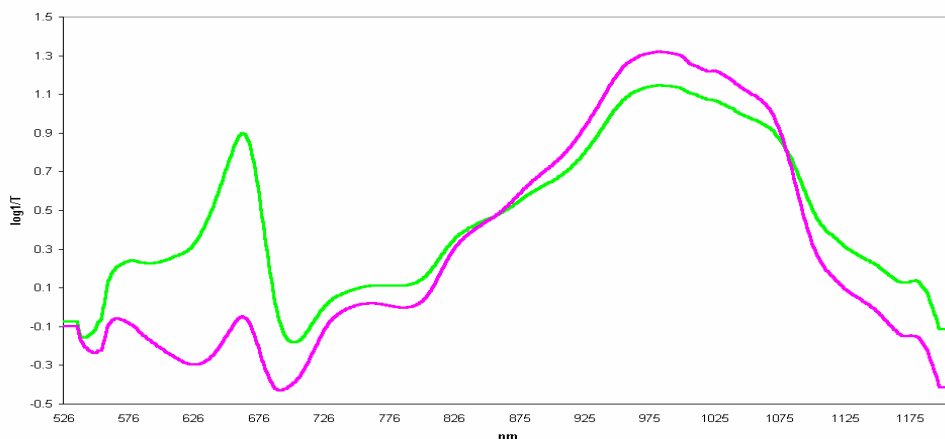


Figure 3. NIR spectra of the three main sugars present in the usual honey composition

The spectra of glucose, fructose and sucrose presented in figure 3 allow to see what are the differences of spectral profile which are exploited in the multi-linear calibration of the spectrosensor to determine the proportions of the three sugars in honey.

The performance estimators of calibration for the determination of water, protein and oil in wheat and the determination of glucose, fructose and sucrose in honey have been computed. Their values were in every case more than 0.9 for the correlation coefficient  $R$  and less than 10% for the relative error equal to the ratios  $SEC/\bar{Y}$  and  $SEV/\bar{Y}$  multiplied by 100,  $\bar{Y}$  being the mean concentration of the analyzed constituent in the calibration samples.

The results of calibration for the determination of sugar in honey demonstrate that the spectrosensor can distinguish very similar molecules such as glucose, fructose and sucrose and even determine their concentration when they are mixed in aqueous solution like honey. Effectively, the spectrosensor has been calibrated with success for a sugar isomerisation process control in a factory of glucose and fructose syrups produced from cereals. The precision of the spectrosensor in the determination of the two monosaccharids during the isomerisation process is equal to the precision obtained in honey analysis.



**Figure 4.** Spectra of a ripe sound apple and an overripe mealy apple.

The spectra of a sound and a mealy apple in figure 4 show large differences of profile between the wavelengths 600 and 700 nm at the end of the visible part, between 900 and 1100 nm, and between 1100 and 1200 nm in the near infrared part. So important differences in three different spectral locations can be exploited easily by the multi-linear equation  $Y = a_0 + b_1x_1 + b_2x_2 + \dots b_px_i$  ... in calibration of the spectrosensor for the sorting of apples according their internal quality. This advantage of the transmittance measurement through the whole fruits has been confirmed by a relative error of calibration  $100(SEV/\bar{Y}) < 10\%$  for the prediction of mealiness.

## Conclusion

If the calibration precision of the NIR spectrosensor is estimated by less than 10% of relative error not only for the experiments presented here above, but also for many other applications of fast and non destructive chemical analyses tested for the food industry in our laboratory (Meurens and Yan, 2001), we can affirm that this instrument is an interesting tool of process and quality control for a lot of enterprises producing and transforming natural, biological and chemical products.

## References

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2. B. G. Osborne, T. Fearn and P.H. Hindle, In Practical NIR Spectroscopy, 2<sup>nd</sup> Edition, Longman Scientific & Technical, Harlow, UK (1993), 99-119.