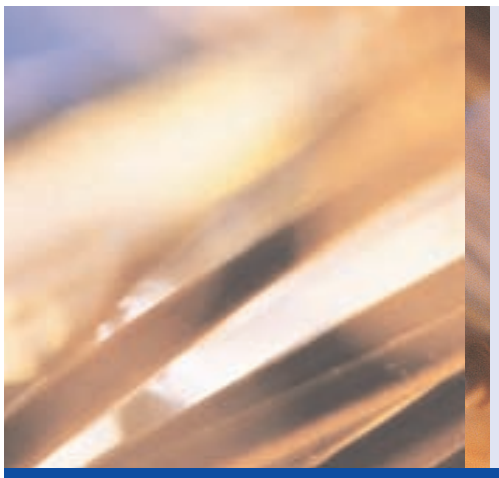

Spectroscopy Focus

NEAR INFRARED TRANSMISSION SPECTROSCOPY IN THE FOOD INDUSTRY

Phil Clancy, NIR Technology Systems

Near Infrared Spectroscopy is used in many industries including the pharmaceutical, petrochemical, agriculture, cosmetics, chemical and food industries.

However in the food industry NIR has an almost universal application. Since food is made mostly from proteins, carbohydrates, fats and water, i.e. >99% by weight, NIR provides a means of measuring these components in almost any food.

"ANOTHER ADVANTAGE OF MEASURING IN TRANSFLECTANCE AS COMPARED WITH REFLECTANCE IS THAT THE SPECTRA REPRESENT THE VARIATION IN COMPONENTS THROUGHOUT THE ENTIRE SAMPLE, NOT JUST THE SURFACE"

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NIR ANALYSIS: SIMPLE THEORY

Part of the electromagnetic radiation spectrum from 300nm to 10000nm covers the Visible, NIR and Mid IR spectral regions. When light energy interacts with a material, energy is absorbed at resonant frequencies associated with the atomic and molecular interactions of the material. In the Visible region, ie, 400 to 680nm, energy is absorbed when electrons jump from a lower to a higher energy state or orbit. In this region, chromaphores such as metal chelates, ionic salts and some organic coloured compounds absorb visible light. Likewise Atomic Absorption Spectroscopy also uses this phenomenon to measure metals species as they burn in a flame.

In the Mid Infrared region, i.e. 2500 to 10000nm, energy is absorbed by vibrating molecules at resonant frequencies for each type of vibration, eg, stretching, bending, wagging, twisting etc. In the Mid IR region, all chemical bonds in organic molecules exhibit strong absorption bands. Typically the Mid IR region is suitable for characterising C-C, C-O, C-N, C-H, O-H, N-H and many other chemical bonds. Sample preparation such as dissolving in a solvent, preparing a nujol mull, drawing film etc, is required in order to get sufficient light through a sample. Mid IR spectroscopy is generally used for non-water bearing materials such petrochemicals, plastics, polymers and chemicals. Mid IR spectroscopy is used mainly for qualitative analysis rather than quantitative analysis.

The NIR spectral region, i.e., 720 to 2500nm, is the Overtone and Combination region of the Mid IR region. NIR spectra contain absorbance bands mainly due to three chemical bonds, i.e., C-H (fats, oil, hydrocarbons), O-H (water, alcohol) and N-H (protein). Other chemical bonds may exhibit overtone bands in the NIR region, however they are generally too weak to be considered for use in analysis of complex mixtures such as foods, agricultural product, pharmaceuticals, toiletries, cosmetics, textiles etc.

NIR spectra do not have the resolution of the Mid IR spectra however NIR spectra can generally be collected off or through materials without sample preparation. As well the NIR spectral region is suitable for measuring high and low water content materials. Whereas Mid IR is mainly a qualitative technique, NIR is mainly a quantitative technique. NIR provides a very rapid means of measuring multiple components in foods, agricultural products, pharmaceuticals, cosmetics, toiletries, textiles and virtually any organic material or compound.

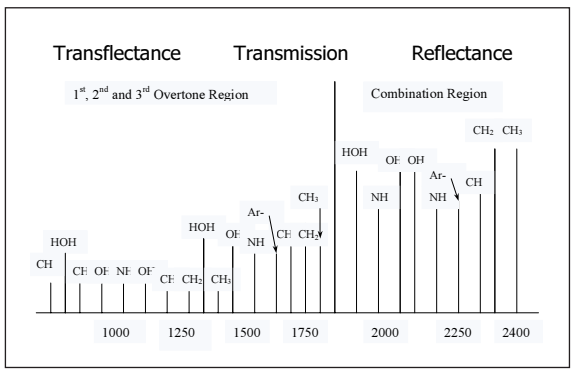

Figure 1. shows the NIR region from 720 to 2500nm. There are three parts of the NIR spectral region, 1) Reflectance, 2) Transmission and 3) Transflectance.

THREE NIR REGIONS

1) Reflectance: 1850 to 2500nm. This section is predominantly used for making diffuse reflectance measurements off ground or solid materials. The absorption bands are due to combination bands, i.e., combinations of C-H stretch and bend vibrational bonds.

2) Transmission: 1200 to 1850nm. This section can be used for transmission through liquids and films, as well as for diffuse reflectance measurements off samples with high water contents. The absorption bands are due to the 1st and 2nd overtones of the fundamental stretch bonds in the Mid IR region.

3) Transflectance: 720 to 1100nm. This section is most suited to transflectance through a thick sample, such as, seeds, slurries, liquids and pastes. The absorption bands are due to 3rd overtones of the fundamental stretch bonds in the Mid IR region.

The Transflectance region is of particular interest in the analysis of foods because it is suitable for measuring high moisture and high fat content products including meat, dairy products, jams and conserves, dough and batters. The major advantage of working in this region is that longer pathlength sample cells can be used to collect the NIT spectra. Typically a 10-20 mm pathlength can be used. This makes sampling easier and allows viscous and non-homogeneous samples to be scanned without further sample processing. Another advantage of measuring in Transflectance as compared to Reflectance is that the spectra represent the variation in components throughout the entire sample, not just the surface. In Reflectance, the first 1mm contributes as much as 99% of the spectrum. As such uneven distribution of components in the sample, e.g., drying at the surface, or separation of a water or oil layer at a glass window, results in reflectance spectra that do not represent the entire sample. Transflectance spectra represent the front and back surface as well as the material in the middle.

The following are examples of NIT analyses of food products using the Transflectance region;

RAW AND PROCESSED MEATS:

Figure 2 shows the NIT spectra of minced beef, minced ham, pork sausage, salami and bologna. These spectra were collected using a 10 mm pathlength Squeeze Cell in which the meat is compressed between two glass windows to make a 10mm thick slab. The spectra in *Figure 2* show significant differences in optical density which cause large variances in the baselines for each spectra. By converting the spectra with a 2nd Derivative or Multiplicative Scatter Correction(MSC) algorithms, these baseline differences are reduced and a more linear calibration can be developed.

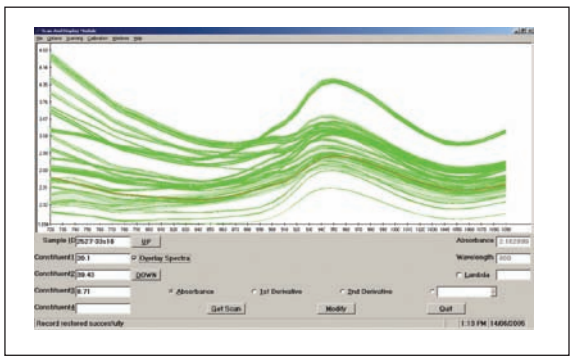

Figure 2. NIT Spectra of various

As an example of quantitative analysis of meat products, *Figure 3* shows the plot of NIR Fat vs Soxhlet Extracted Fat. A calibration for fat was developed using 79 samples and a further 21 samples were analysed in duplicate to define the accuracy and precision. The accuracy or Standard Error of Prediction(SEP) was calculated as 0.7% fat and the precision or Standard Deviation of Difference(SDD) was calculated as 0.19%. Calibrations for protein, moisture and salt have also been developed for processed meat samples.

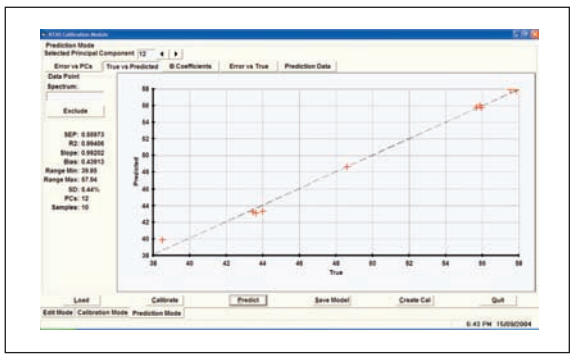

Figure 3. Plot of NIR Fat vs Soxhlet Extracted Fat calibration data.

DAIRY PRODUCTS:

Figure 4 shows the NIT spectra of milk, cheddar cheese, yogurt, milk powder, cream cheese and butter. Except for milk powder, these spectra were also collected using a 10mm pathlength Squeeze Cell. The milk powder was scanned in a 5mm pathlength Powder Cell. Typically fat, protein and moisture are the components of interest in dairy products, although lactose is measured in milk. The accuracy of the NIR techniques depends on the reference method to which the NIR method is compared. For fat the expected SEP would be between .1 and .5%, for protein, between .1 and .3% and for moisture, between .1 and .5%.

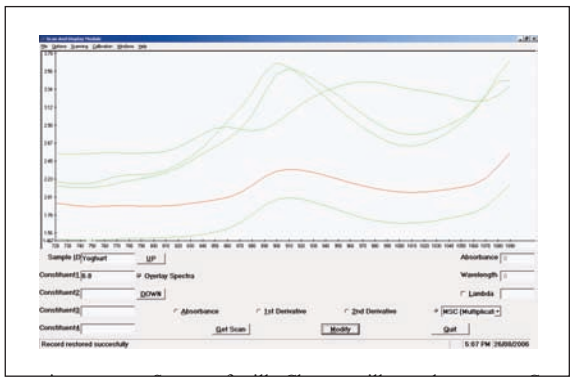

Figure 4. NIT Spectra of Milk, Cheese, Milk Powder, Yogurt, Cream Cheese

BAKED GOODS

Figure 5 shows the NIT spectra of cookie dough scanned in a 20mm pathlength Squeeze Cell. Fat, moisture, protein and sugar can be measured in dough, however the ability to rapidly measure fat and moisture in the mixing room, means that the dough will spread correctly when the cookies are laid on the hot oven band. If the fat content is too high then the cookies will spread too much, resulting in out of spec product. The accuracy of the fat and moisture measurements has been shown to be 0.13% and 0.28% respectively.

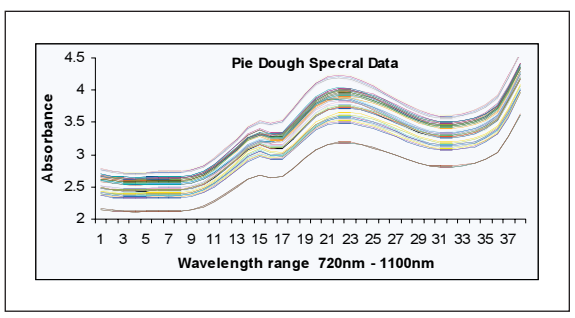

Figure 5. NIT Spectra of Pie Dough

In the baked good industry, NIT can also be used for measuring the incoming flour for protein, moisture and ash, as well as dough stability, dough development and water absorption. Other raw materials used in baking that can be analysed using the NIT spectral region include, liquid and powdered eggs, milk and milk powder, shortening, liquid and crystalline sugars and syrups.

CONFECTIONERY

Chocolate is the world's most popular confectionery. *Figure 6* shows the NIT spectra of a milk chocolate and a dark chocolate using a 5mm pathlength Squeeze Cell. The chocolate samples were softened but not melted. The softened block was compressed between two glass windows.

Reflectance spectra of chocolate suffer from the problem of drying and uneven distribution of the fat at the surface and the rest of the sample. Transflectance provides a quick method of measuring fat and moisture throughout the entire chocolate sample.

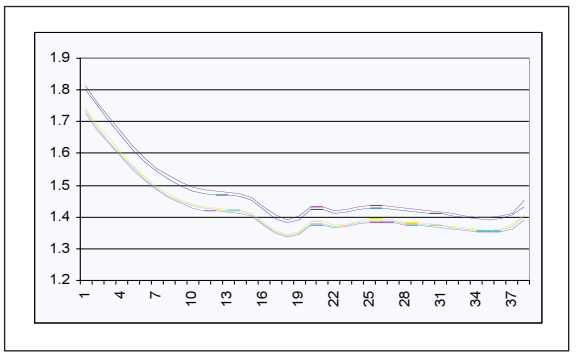

Figure 6. NIT Spectra of Milk and Dark Chocolates

Figure 6 also shows the NIT spectra of several common types of lollies and candies. Sweets such as jubes or jellies, are made of gelatin, i.e., protein, water and sugar. Since these sweets are easily deformed, they can be measured in a 10mm pathlength Squeeze Cell.

Nougats and caramels used in many candy bars are a mixture of fat, sugar and emulsifiers. NIT spectra are easily collected and as such provide confectionary manufacturers a rapid means of measuring the consistency of the material before it is used in the final bar.

GRAINS AND OIL SEEDS

The Transflectance region(680-1200nm) was first suggested by Karl Norris as a means of measuring whole cereal grains and oil seeds because the NIR light could penetrate through the grains and oil seeds. Actually the Transflectance mechanism is a combination of reflectance and transmission, as the NIR light reflects off the surface of the seeds as it transmits through the sample to the other side.

Figure 7 shows a schematic of how light passes through a sample of wheat. *Figure 8* shows the NIT spectra of wheat, barley, soy beans, corn (maize) and canola seeds. It is difficult to measure whole seeds in the Transmission and Reflectance regions because of the large amount of light absorbed by the sample .

However in the Transflectance region, the NIT spectra can be collected using a long pathlength. This makes sampling very easy and lends itself to on line measurements.

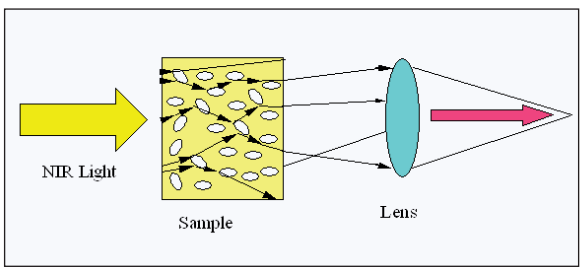

Figure 7. Schematic of how NIR light interacts with solid particles in the Transflectance Mode

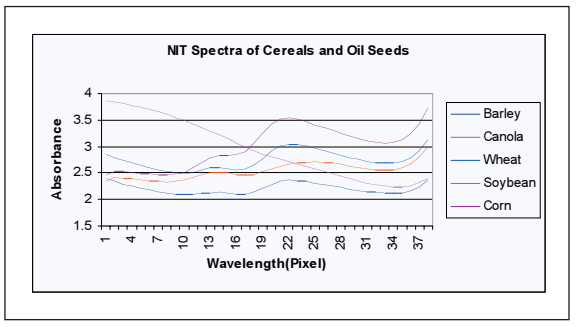

Figure 8. NIT Spectra of Cereals and Oil Seeds

BEVERAGES

Wine, Beer, Spirits and even alcoholic mixer drinks, can be analysed for alcohol using NIT. *Figure 9* shows the NIT spectra of several alcoholic beverages. Other non-alcoholic beverage scan also be analysed by NIT, such as soy milk, fruit juice and yakult. Refractive index is a low cost method for sugars whereas NIT is capable of measuring sugar, protein, fat and solids in the same sample.

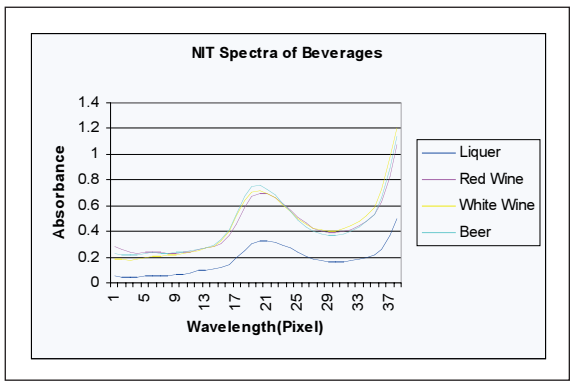

Figure 9. NIT Spectra of Beverages

CONCLUSION

Near Infrared Transmission Spectroscopy makes the analysis of many complex foods look simple. Although all NIR methods require calibrations to be developed for each sample matrix and as such, there are no standard or off the shelf calibrations for most foods, the speed and simplicity of NIT methods will make the effort of calibration worthwhile.