Using MicromATR Vision™ for Food Analysis: Measurement of the Macro-Nutrients

Introduction

Infrared (IR) spectroscopy has been used for nearly fifty years for the analysis of food for macro nutrients. In this case, the term macro nutrient refers to fats, proteins and carbohydrates. Near infrared (NIR) in recent years has also been used based on the simplicity of sample handling, and the use of diffuse reflectance. However, in order to obtain a quantitative measurement from the NIR spectral data it is necessary to build and maintain chemometric models. Furthermore, the primary functional groups associated with these nutrients, such as carbonyl groups linked to fats and proteins, and ether (C-O-C) groups linked to carbohydrates are directly measured in the mid-IR (4000-400 cm-1). As an easy to use attenuated total reflectance (ATR) system, the MicromATR Vision provides the simplicity of sample handling of an NIR based measurement while providing a direct measurement of the macro nutrients in the mid-IR. A wide range of sample interface options (disks) are available for MicromATR Vision. These options may be required to address specific chemical properties of analyte materials and effective pathlengths. All three nutrients can be observed as discrete absorptions and these may be measured and quantified without the need to build and maintain chemometric models. This application note illustrates the point by providing spectral examples from the measurement of the sugar content of grapes (grape juice) to the measurement of fats in cheese, chocolate and milk.

Determination of Sugar and Acid Content of Fresh Grape Juice

IR spectroscopy has been used for process monitoring in the beverage industry for the measurement of carbon dioxide in soda drinks and for the sugar content. An example application, shown below in Fig. 1, features the measurement of the sugar content of grapes, where grape juice is squeezed out of the grape onto the surface of a 9 reflection MicromATR sample interface. The juice spectrum was ratioed against the spectrum of water. This removes the contribution of water, the major component of the juice, and this provides a clearly defined spectrum of the sugar component of the juice with the major absorption (C-O-C) indicated centered at 1064 cm-1. The profile of this absorption can be correlated with the main sugar components, which are expected to be glucose and sucrose. A minor absorption band at 1758 cm-1, supported by the broad band between 3200 cm-1 and 2400 cm-1, and centered at 2933 cm-1, is correlated to the free acid content of the grapes juice, with tartaric and malic acids being the major acid components. The sugar and acid composition are both important attributes of grape juice relative to wine production. While sugar content may be estimated by BRIX value, as measured by refractive index, and full compositional analysis obtained by IR spectroscopy provides a more detailed result.

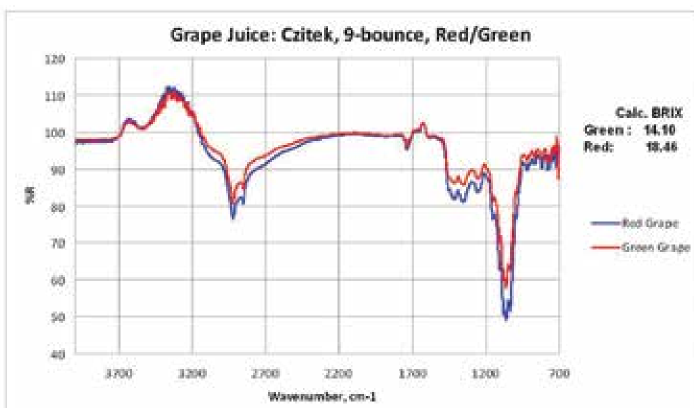

Figure 1: Spectrum of fresh grape juice from a squeezed grape - 9 reflection Cleartran ATR head

Measurement of the Fat Content of Cheese

Fat in dairy products was originally determined in early instruments from the C-H absorptions, nominally at 2926 cm-1 and 2854 cm-1. These can be seen clearly in the spectra in Fig. 2, which illustrates the ATR spectra for isolated milk fats, and samples of cheese as measured on the single reflection diamond sampling head. These C-H bands are observed on the side of a broad absorption, primarily linked to hydroxyl (O-H) and amino (N-H) bands, which vary as a function of overall composition. Water (a major component in milk) and other hydroxyl compounds such lactose and other sugars, and amino and protein all contribute to this complex absorption. Another issue is that most nutrient components have C-H functionality, and so the C-H absorptions are not unique to the fats.

A good alternative for the determination of fat content is the well defined C=O band (1743 cm-1) from the fat lipids (esters). A detailed look at the C=O band of a range of cheeses is provided in Fig. 3. The good news about this measurement is that other C=O components may be observed, and these can be correlated other carbonyl containing components, including fatty acid components, which are characteristic components of many cheeses. Measurement of the main fat lipid absorptions can be differentiated from the other carbonyl compounds by the use of spectral band deconvolution functions, including the use of a simple 2nd derivative computation.

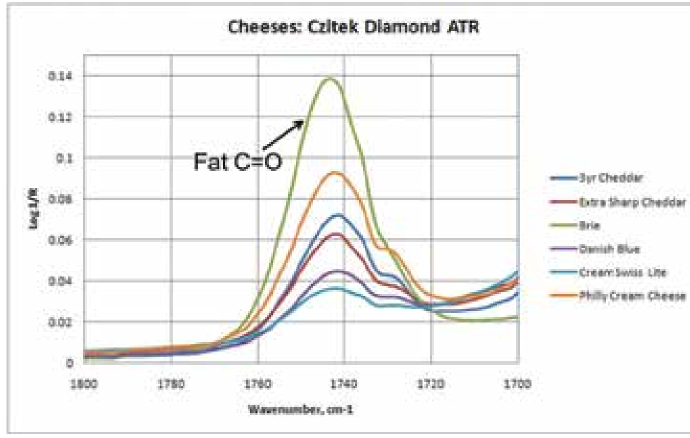

Figure 3: Measurement of fat in cheese and cheese products

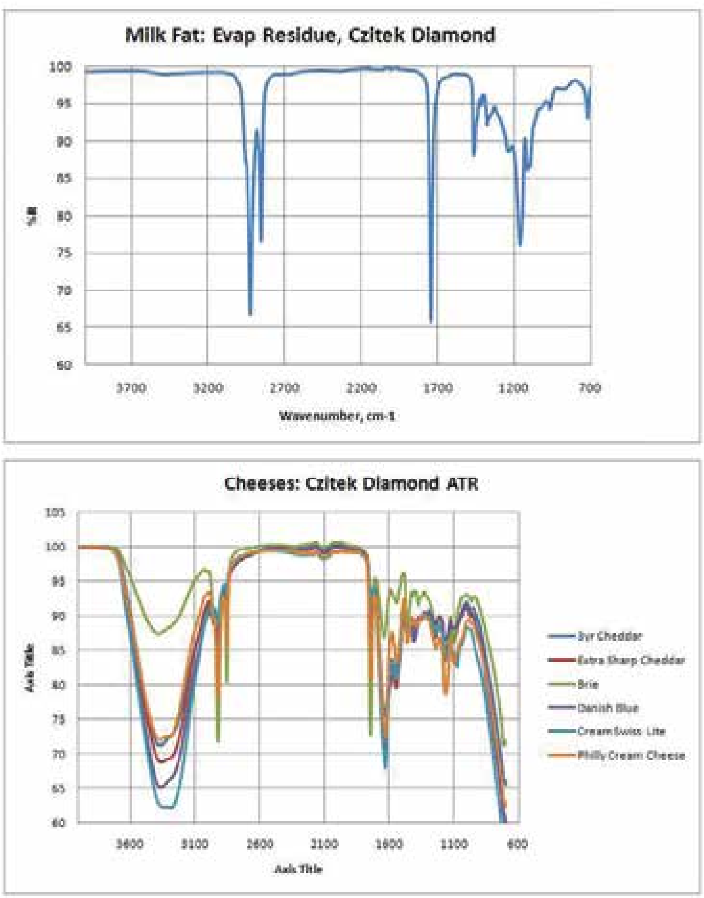

Figure 2: Milk fat and the fat content of cheeses

Fat and Sugar Content of Chocolate

Chocolate is a complex and interesting material to study by IR spectroscopy, and the MicromATR is an ideal tool to use. The final product exists in various polymorphic forms, and the crystal structures, six different forms exist, define the physical properties of the chocolate…and these have a big impact on how consumers perceive the product. Commercially, there are three common forms of chocolate products: dark chocolate, milk chocolate (light chocolate) and white chocolate. Dark and milk chocolate are covered in this note, and part of the spectral data is shown in Fig. 4. The main ingredients are noted below:

Dark: cocoa solids (cocoa powder), cocoa butter, sugar and sometimes vanilla

Milk: cocoa solids, cocoa butter, sugar, milk or milk powder

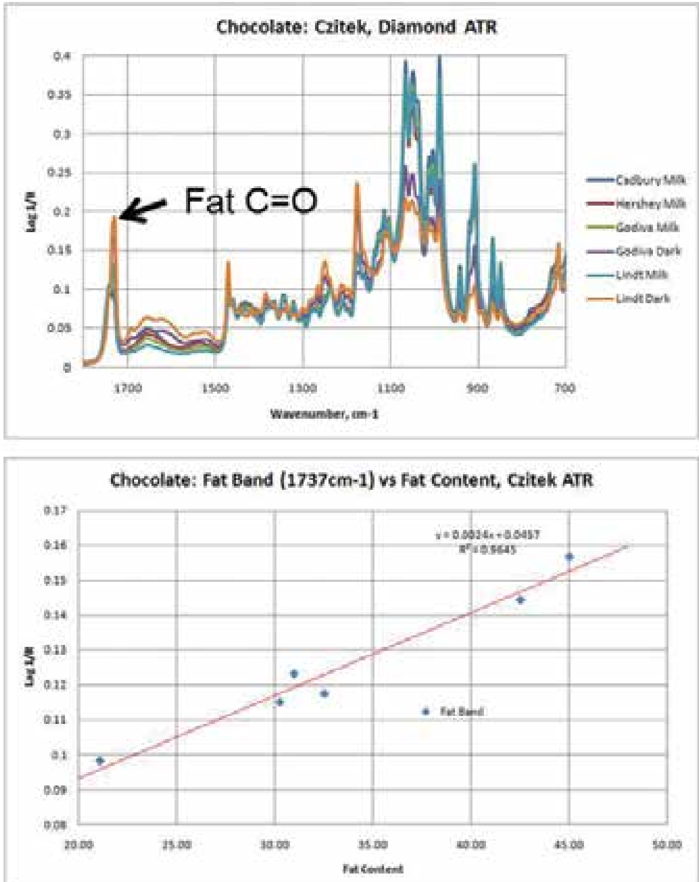

Figure 4: The determination of the fat and sugar content of chocolate (fat response curve shown)

The cocoa butter is a major source of fat in the product, and this is a triglyceride primarily composed of palmitic and stearic (saturated fats) and oleic (mono unsaturated) acid esters. The cocoa solids provide carbohydrates, some residual cocoa butter fats and protein to the product. As a simple exercise, the fat content of the chocolate, as recorded on the bar wrapper, is plotted against the intensity of the intensity of the 1734 cm-1 absorption. Similar quantitative responses can be observed for sugar content by measuring the complex sugar absorption bands located between 1100 and 800 cm-1.

This applications note is intended to only indicate the potential of IR spectroscopy for the characterization and quality assessment of this important and popular food group. The complexity of the IR spectrum and the information that can be obtained justifies an applications note focused on chocolate. This will be covered in a future publication.

Proximate Analysis of Liquid Milk Products

Milk has been a subject of study for the infrared measurement of macro nutrients, fats, proteins and sugars for decades (since the 1970s). This measurement is sometimes referred to as the proximate of liquid milk products. The initial application was covered by the use of a short pathlength (approximately 0.04 mm) transmission cell. This imposes a physical complication on the measurement because of the difficulty of filling and cleaning the cell. In commercial analyzer systems this has been handled by in-line homogenization, with heating (~40°C) and pressure pumping through the cell.

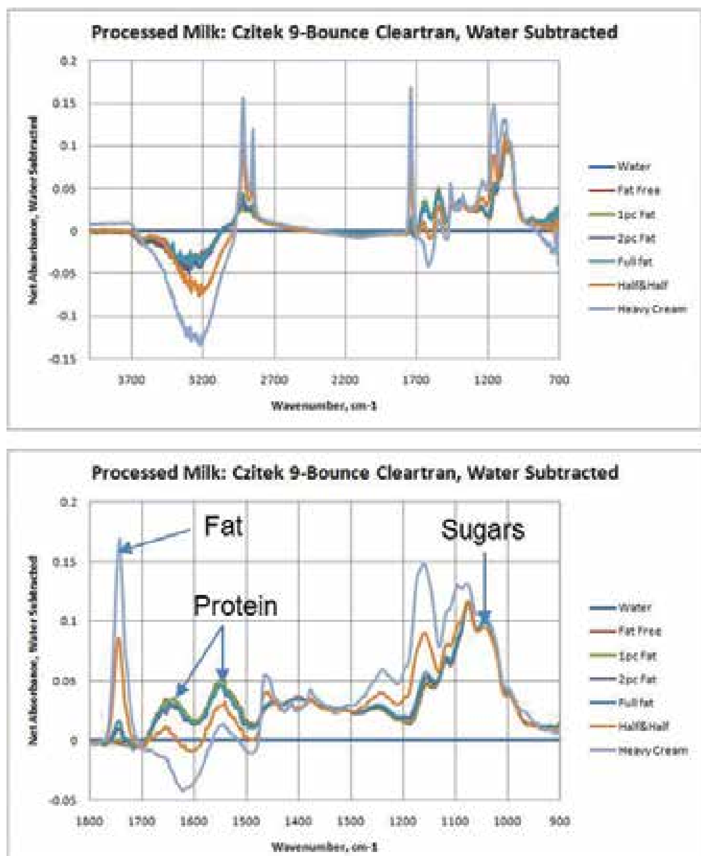

Figure 5: Proximate analysis, fats, protein and lactose of liquid milk products

As a sampling method, ATR provides a good solution for this application that overcomes the physical limitations of a short pathlength transmission cell. Fig. 5 illustrates this point by showing the spectrum of a series of liquid milk products – fat free (nominal 0% fat) to heavy cream (nominal 33% fat). The spectra as shown have been ratioed against the spectrum of water, the main component of liquid milk. These spectra were recorded by the use of a 9 reflection ATR head and in this case the ATR material used was Cleartran (a refined form of zinc sulfide, ZnS). Diamond or Cleartran reflection elements may be used for this application. The primary benefit of the Cleartran is that it does not have strong diamond background absorption that partially overlaps the fat band. The main ingredients required for the proximate analysis are shown above in the spectra in Fig. 5. As observed for this processed milk sample series, the main composition variation noted is the fat content, while the nominal protein and lactose contents remain more or less the same. This is intended as an example for the application, and as a practical method the overall analysis can be used for milk production and final product analysis, from the farm to the super market…and to the breakfast table.

Conclusion

This application note has been intended to be a "taster" (no pun intended) to illustrate the role of ATR in the analysis of food and beverage products for the composition and analysis of the main macro nutrients. A more detailed study will be provided at a later date, and this will demonstrate how much valuable information is locked up in the IR spectrum of a food/beverage product. The MicromATR is a critical tool for the unlocking of this information.