Introduction

Dairy powders are used extensively as functional ingredients in a myriad of food products. The composition of dairy powders can be highly variable in terms of proteins, carbohydrate (normally lactose), fat and moisture content. The characterization of dairy ingredients is traditionally carried out by solvent extraction of the fat, followed by chromatographic separation of the proteins and lactose. While these methods are highly accurate, they are time-consuming, tedious and costly. In recent years vibrational spectroscopy has been proven to be an ideal way to analyze powders, giving results within minutes of sample acquisition without prior sample preparation. Among the various vibrational techniques available: Fourier transform infrared (FTIR), near infrared (NIR), and Raman spectroscopy, mid-FTIR spectroscopy appears to be the most useful.
This is because mid-FTIR spectroscopy can provide a great deal of structural information from spectra characterized by very sharp spectral bands that can be assigned to specific functional groups, thereby making mid-FTIR spectroscopy a highly valuable technique for the identification of complex dairy ingredients. Furthermore, using an attenuated total reflectance (ATR) sampling accessory in combination with an FTIR spectrometer, as in the case of the Cary 630 ATR-FTIR analyzer, provides the best means for routine authentication of dairy powders and quality assurance/quality control (QA/QC) applications aimed at assessing the batch-to-batch variability of powder ingredients.

Objective

Government regulatory agencies and consumer groups are increasingly demanding that the food industry authenticate all raw materials going into their products. To address such a challenge in a timely and cost-effective manner, product manufacturers usually require an advance sample of food ingredients be shipped to them for testing prior to accepting a large quantity of the commodity. However, once the shipment arrives there is still the question of whether the advance samples were indeed representative of the bulk shipment and there is also a need to confirm that the bulk shipments are uniform in composition. For example, since most dairy powders to the naked eye appear white, with a soft grainy texture and a virtually identical structural consistency (Figure 1), it is necessary to utilize analytical methods to characterize their composition.

Figure 1. Various milk protein powders

Ideally, this analysis should be completed in less than 30 seconds, require little or no sample preparation and should be carried out at the receiving dock prior to placing the dairy powder ingredients into storage or integrating them directly into the production line.

In this application note, α-lactalbumin, an expensive whey protein, must be uniquely differentiated from other dairy ingredients such as β-lactoglobulin, whey protein isolate (WPI), whey protein concentrates (WPC) or even other types of dairy proteins such as caseins or caseinates.

Experimental

Materials and instrumentation

• Various milk protein powders obtained from different suppliers including α-lactalbumin, β-lactoglobulin, glycomacropeptide, milk protein concentrate, WPI, WPC, caseins and caseinates.
• A robust and light-weight Agilent Cary 630 ATR-FTIR analyzer (Figure 2).

Figure 2. Agilent Cary 630 ATR-FTIR analyzer

Method

Spectral acquisition was carried out by:

1. Placing a small amount of protein powder on the diamond ATR surface.

2. Pressing the samples against the diamond crystal using the attached pressure clamp. A slip clutch on the clamp prevents overtightening.

3. Collecting 64 co-added spectra (~30 seconds acquisition time at 4 cm⁻¹ resolution) between 4000 and 650 cm⁻¹.
Data processing
A database of spectra of representative dairy powders was constructed using the Agilent MicroLab FTIR software spectral library builder. Subsequently, additional spectra of different lots of dairy powders were recorded and treated as ‘unknowns’. For example, the ATR-FTIR spectrum of α-lactalbumin was acquired and treated as an ‘unknown’ and was identified correctly as α-lactalbumin through the automated spectral matching feature of the MicroLab FTIR software (Figure 3).

Results and discussion

Figure 4 shows typical infrared spectra acquired using the Cary 630 ATR-FTIR analyzer for α-lactalbumin, β-lactoglobulin, WPI, and WPC. The difference between the other three proteins and WPC (attributed to the presence of lactose in WPC) can be clearly seen in the presence of additional bands between 1300 and 900 cm\(^{-1}\). The differences among the three other proteins are not as easily distinguishable by the naked eye, but by utilizing the built-in spectral analysis MicroLab FTIR software, all four protein types can be immediately characterized and differentiated.

Conclusion
The Agilent Cary 630 ATR-FTIR analyzer can dramatically decrease the cost and time for analysis of dairy powder ingredients with minimal training and virtually no sample preparation. The analyzer is compact, lightweight and portable making it ideal for use at the receiving dock, in production lines or in the QA/QC laboratory.

Suggested references

