FTIR analysis provides rapid QA/QC and authentication of food ingredients prior to processing

Application note

Food testing and agriculture

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Introduction

The food industry is increasingly focused on the analysis, authentication and characterization of the raw materials and ingredients that are used in foods, in addition to analyzing the quality of the final food product. This is driven by regulatory agencies, consumer groups and the overall demand for safer foods, especially given the impact of globalization of sources. From a manufacturer's perspective, the goal is to make sure that substandard, adulterated or incorrectly labeled ingredients never enter the manufacturing process. Ideally, this check-point happens directly at the loading dock, the truck stop or the rail car.

New analytical tools are needed that provide equivalent analytical capability as lab instruments, yet are sufficiently robust and intuitive to be used closer to the source of the sample by less experienced persons.

In a recent series of application notes, we demonstrated that a small robust lab FTIR, the Agilent Cary 630 Spectrometer, is very effective at analyzing, characterizing and verifying raw materials and ingredients used in foods (1-4).

Figure 1. Cary 630 FTIR spectrometer - for the QA/QC laboratory

In addition to the lab-oriented Cary 630 spectrometer (Figure 1), Agilent offers the Agilent 4500 and 5500 FTIR analyzers, which use the same optics and innovative sampling technology as the Cary 630, yet are purpose-built for out-of-the-laboratory applications, and closer to the source of the sample.

Figure 2. 4500 FTIR analyzer – battery powered, for analyses at the loading dock, tanker car, etc.

The Agilent 4500 system (Figure 2) is a self-contained battery operated analyzer that can be used in the field, at the truck stop or next to the railroad tanker. The Agilent 5500 system (Figure 3) is well suited for at-site labs or other demanding locations where power is available.

Figure 3. 5500 FTIR analyzer – for analyses in at-site labs

The Cary 630 is a multi-purpose FTIR spectrometer, which is ideal for the traditional analytical, QA/QC or methods development lab. All three spectrometers provide identical results.

This note reviews the application of FTIR spectroscopy to the analysis of an assortment of powdered food ingredients utilizing state-of-the-art instruments such as the Agilent Cary 630 FTIR, and the Agilent 4500 and 5500 FTIR analyzers, each of which are available with the identical innovative ATR sampling technology (Figure 4). The powders analyzed for authenticity include milk proteins, sugars, coffee and flours.

This choice of systems allows users to decide whether their manufacturing processes require an analyzer that is used in the lab or at the sample site while ensuring that whatever the choice, the quality of data and answers are identically superior.

Figure 4: Agilent single reflection diamond ATR sampling technology for Cary 630 (pictured), 5500 and 4500 systems is ideal for measuring powdered food

Advantages of the Agilent FTIR Spectrometers

Agilent Cary 630, 4500 and 5500 FTIR analyzers are extremely well suited to analyzing raw material and ingredients used in the food industry for the following reasons:

- They provide answers in less than 30 seconds, do not require sample preparation, and use no consumables, making the analysis far simpler than other techniques.
- These systems are mid-infrared spectrometers and thus provide far more specific detail and insight into the composition of a food sample when compared to the historically, widely used NIR spectroscopic method.
- They are the smallest FTIR spectrometers currently available and thus are readily transportable, taking up very little space on crowded lab benches or on loading docks.
- These exceptionally robust systems use proven optomechanical technology that is engineered to easly meet the rigorous demands of the out-of-lab environment, regardless of location.
- These analyzers combine class-leading performance with unsurpassed reliability. This means that lower level impurities can be more effectively detected, as well as ensuring the accuracy of acquired data.
- Measuring samples using the Agilent diamond ATR interface means that no sample preparation is needed – simply place the sample on the sensor and use the sample press to ensure good contact. The sensor is made of diamond, so there is no concern that the sample can abrade or scratch the sensor surface.
- These systems require minimal maintenance; no alignment or re-calibration is required.
- The systems use Agilent's Microlab software – this highly visual, intuitive software allows even less experienced users to get great data from the system. On-board methods run automatically, providing clear accurate answers.

Advantages of moving FTIR analysis from the lab to the point of entry of the raw material

- When a shipment of raw material arrives, FTIR (a zero sample prep technique) rapidly analyzes a statistically representative number of samples of the incoming ingredient. No more guessing whether the sample that was sent to a lab reflects the bulk shipment
- Results are virtually instantaneous, enabling actionable accept-or-reject decisions on the spot.
- Decisions at the point-of-entry ensure that incorrectly labeled, substandard, or otherwise unacceptable ingredients never enter the manufacturing process.
- On-the-spot analyses means no time is wasted waiting for results from a lab that might be either distant or backed up with sample load.
- On-board validated methods that run automatically allow less experienced personnel to get accurate answers.

Application of Agilent FTIR analyzers to the analysis of food ingredients

Materials and Experimental

The powdered raw materials and ingredients analyzed with FTIR spectroscopy include:

- Milk proteins: alpha-lactalbumin, beta-lactoglobulin, glycomacropeptide, milk protein concentrate, whey protein isolate (WPI), whey protein concentrates (WPC), caseins and caseinates
- Sugars: alpha-lactose, sucrose, glucose, mannose, xylose, fructose, thaumatin, levulose
- Coffee: Columbian, Colombian Instant, Decaf Columbian Instant, Greek, Turkish, East Mediterranean, Kenya, French Vanilla
- Flours: chickpea, oat, rice, chestnut, millet, soya, corn, hemp, wheat gluten, whole wheat, breadcrumbs, baking soda. Flour products: biochoix, corn pasta, macaroni, penne quinoa, penne rice bran

An Agilent FTIR analyzer, equipped with diamond ATR sampling technology was employed for these measurements. Total measurement time was approximately 30 seconds (64 co-added scans at 4 cm^{-1} resolution). The ATR employed a single reflection diamond sensor. In all cases, a database of representative powders was constructed using the Agilent Microlab FTIR spectral library builder. Subsequently, spectra of different lots of the powders were measured and considered as unknowns. The spectra of the unknowns were then processed by the Microlab automated spectral matching algorithms, which provided the identity of the unknown and a numerical value indicative of the match quality.

Examples

Milk proteins

The infrared spectra of four different milk proteins are visually similar, the exception being WPC, attributed to the presence of lactose (Figure 5). When processed with the automated matching algorithm of the Microlab software, the identity of an unknown protein sample is rapidly determined (Figure 6).

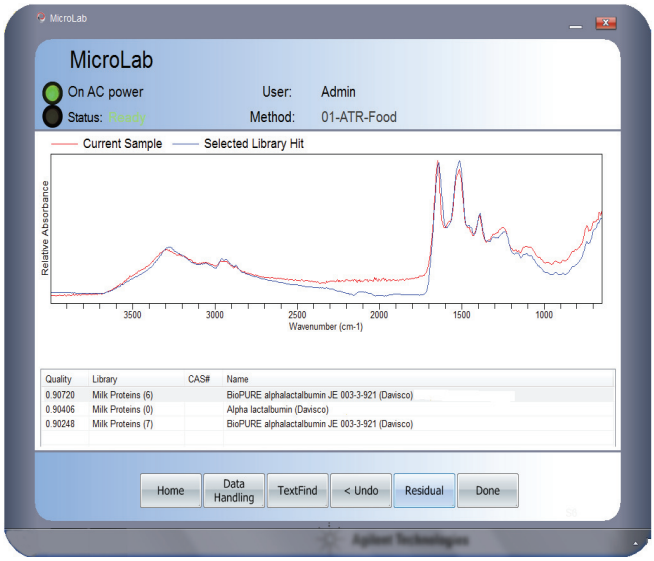

Figure 6. Unknown milk protein is identified by automated matching of its spectra with reference spectra in on-board library

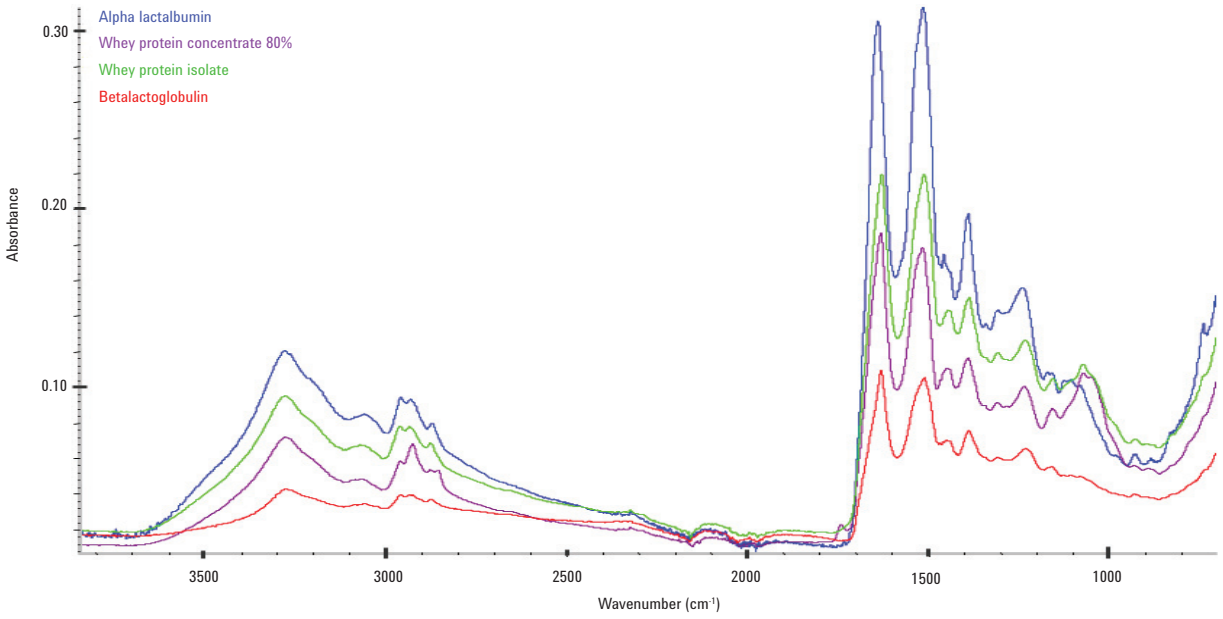

Figure 5. Infrared spectra of milk proteins measured by single reflection diamond ATR

Sugars

The chemical identities of various sugars are nearly impossible to determine visually and currently most manufacturers use chromatographic methods to verify that the correct sugar is used in their production. This method is time consuming and is not amenable to use outside of a laboratory environment.

In contrast, spectra of various sugars (Figure 7) provide clear insight into the identity of a sugar and this is mathematically verified using the spectral library matching capability of the Agilent FTIR analyzers (Figure 8).

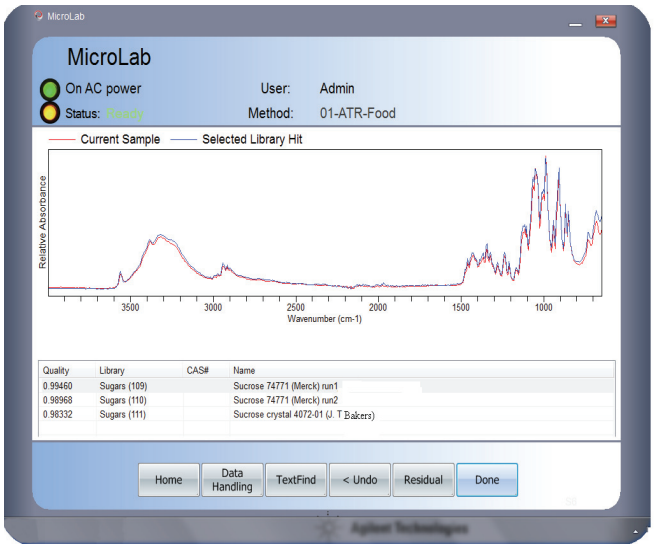

Figure 8. Unknown sugar sample is identified by automated matching of its spectra with reference spectra in on-board library

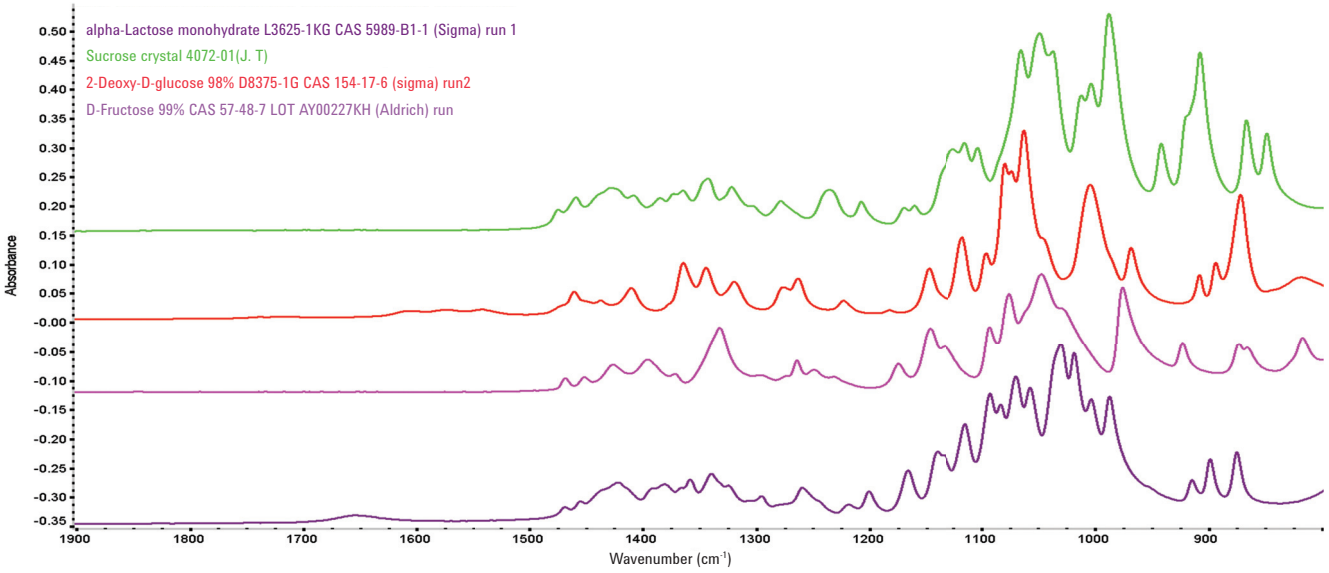

Figure 7. Infrared spectra of sugars measured by single reflection diamond ATR 5

Flours

Flour is typically received as a powder and the identity must be verified to ensure that the received material matches the container label and that the product meets the requirements of uniformity and compositional purity. The infrared spectra of various types of flours (Figure 9) provide detail into the moisture, fat, protein and carbohydrates present and the subtle differences between the spectra of these flours provide the information required for differentiation of identity by spectral library matching (Figure 10).

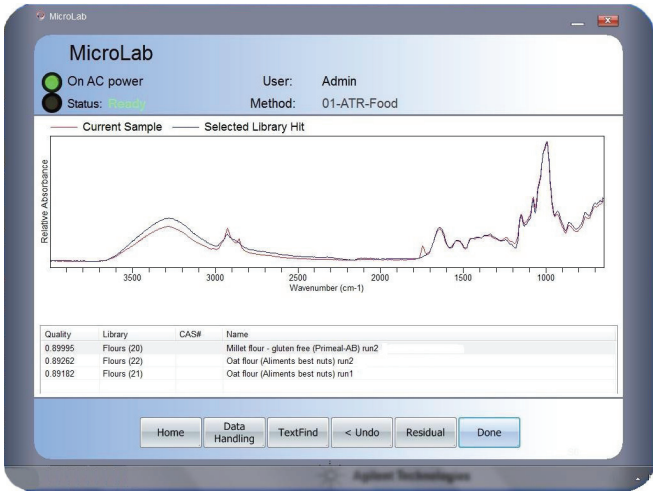

Figure 10. Unknown flour sample is identified by automated matching of its spectra with reference spectra in on-board library

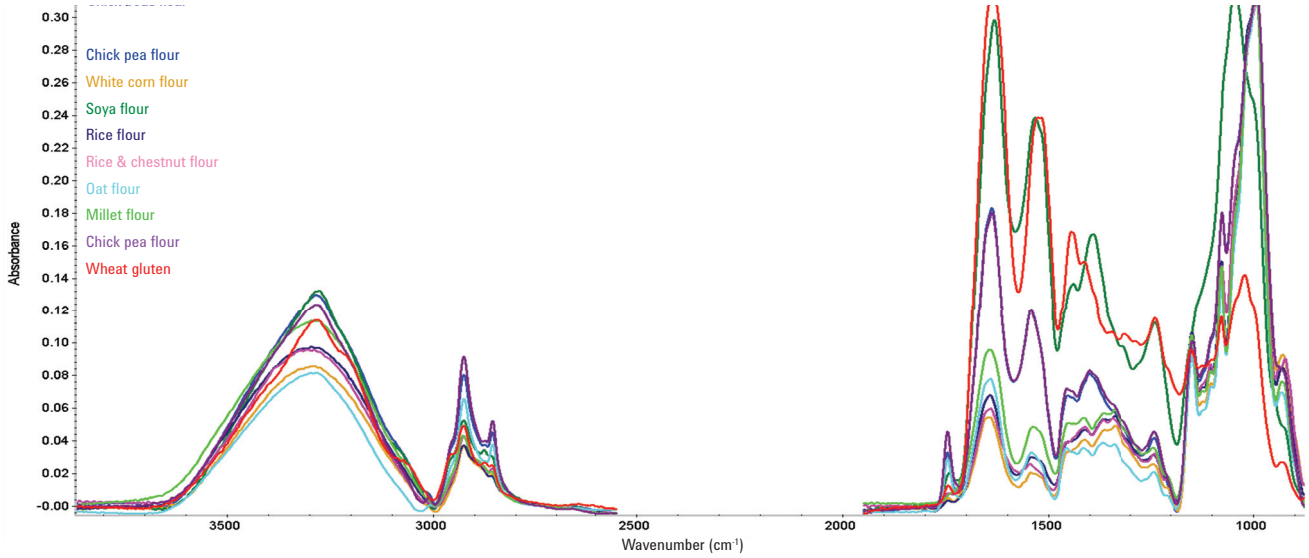

Figure 9. Infrared spectra of flours measured by single reflection diamond ATR

Coffee

Coffee taste is dependent on a number of intrinsic chemical factors, as well as on external influences such as the moisture and temperature levels that the coffee beans are exposed to during shipment. Acceptance of a shipment of the beans requires an analytical method that enables a coffee manufacturer to make on-the-spot decisions. FTIR spectroscopy has proven to be ideal for this purpose, detecting subtle differences in the chemical composition of coffee. The infrared spectra of various coffee powders (Figure 11) show both subtle and distinct differences and the identity of a specific type of coffee is readily determined by spectral matching of the sample with the automated Microlab software (Figure 12).

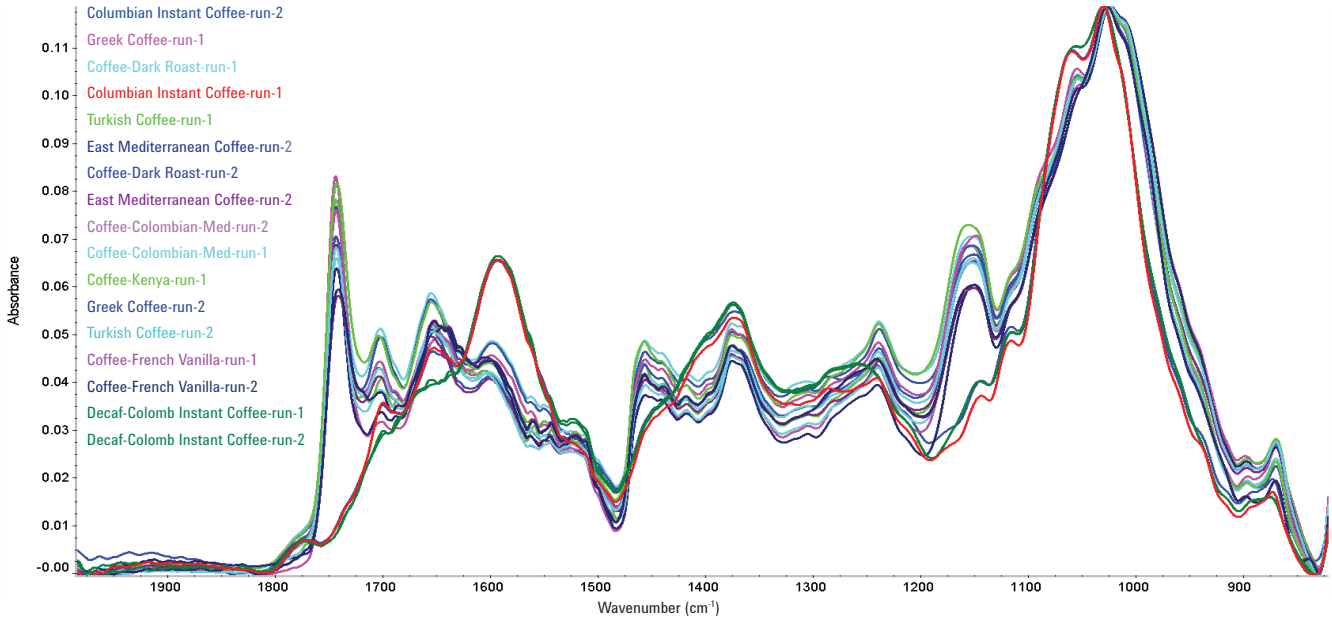

Figure 11. Infrared spectra of coffee measured by single reflection diamond ATR

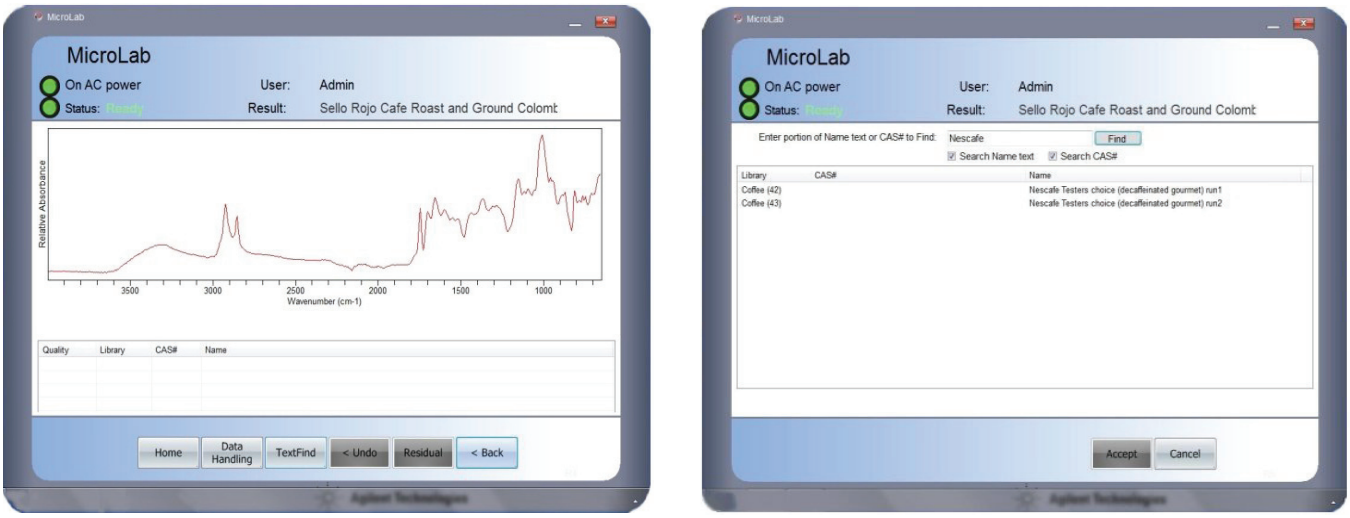

Figure 12. Unknown coffee sample is identified by automated matching of its spectra with reference spectra in the on-board library

Conclusion

FTIR analyzers utilizing ATR sampling technology offer food manufacturers a rapid, easy, means of verifying the identity, authenticity and purity of the raw materials and ingredients that are used in the food industry. Fully portable, field ready FTIR analyzers such as the Agilent 4500 and 5500 systems provide additional benefits because they allow the sample to be analyzed and confirmed at the point-of-entry into the manufacturing process—at the "front gate" of the factory. In this manner, keeping out-of-spec raw materials from entering the manufacturing process supports lower cost production, food safety and overall food quality.

Acknowledgment

We wish to thank Professor Ashraf Ismail, Department of Food Science and Agricultural Chemistry, McGill University for data and information contained in this application note.

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Available from www.agilent.com

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