APPLICATION NOTE

No. 52922

Food quality analysis via cost-effective FTIR microscopy

Key words: ATR, Contaminants, Filtration, Filter Analysis, Food Production, FTIR Microscopy, Particulates

Abstract

Ensuring quality and safety in foods and beverages requires continuous vigilance. It is essential to identify unexpected components to determine if they are contaminants requiring elimination or acceptable byproducts of the food production process. FTIR microscopy is a powerful method for identification of traces of material, such as particulates or oil droplets. The vast majority of analyses involve visible irregularities, setting an approximate lower limit of 25 micron sizes. We present two sets of analyses, one involving liquid filtration and the other examining particulate residues in food items, using a simple-to-operate FTIR microscope and ATR sampling.

Introduction

The food and beverage industry processes massive quantities of material for direct consumption or as an ingredient for further processing. A single milk truck, for instance, may carry about 36,000 liters (around 9,000 gallons) of liquid milk per load and even small dairy operations take delivery from several trucks daily. Food processing of all kinds brings the product into contact with containers, impellers, piping, seals and lubricants.

Dark flecks may originate from overcooked material or other contaminations. Identification and isolation of these variances is a critical part of ensuring the food chain.

Modern processing plants are extremely careful to ensure all surfaces the food contacts are inert, non-toxic and scrupulously clean. Even so, the food may pick up small amounts of material, including micro-particles of polymers from plastic impellers, lubricants, oils needed for production or burned materials from cooking. For many beverages, a significant control barrier involves filtration.

Figure 1: Thermo Scientific Nicolet iN5 FTIR microscope

Efforts to ensure safety in the United States (U.S.) took a large leap in 2011 with significant new regulations. According to the U.S. Food and Drug Administration (FDA) web page:

> The FDA Food Safety Modernization Act (FSMA), the most sweeping reform of our food safety laws in more than 70 years, was signed into law by President Obama on January 4, 2011. It aims to ensure the U.S. food supply is safe by shifting the focus from responding to contamination to preventing it.

For the beverage example, this means the particles on the filter are a major analytical target, vital in improving prevention. The Thermo Scientific™ Nicolet™ iN™5 FTIR microscope, shown in Figure 1, is ideally suited to the analysis of filters and other particulates. The cost per analysis from testing laboratories may exceed $200.00 (USD) each and require two to fourteen days. With many analyses per day, this is slow and grows prohibitively expensive. A single, moderately trained non-specialist can run dozens of samples each day with the Nicolet iN5 microscope, resulting in a cost-effective and timely solution.

In this note, residues on a filter, particulates on a chip bag, and dark material from corn meal are analyzed. No sample preparation was needed for any of these, and each analysis required only minutes.

Experimental

A Nicolet iN5 FTIR microscope attached to a Thermo Scientific™ Nicolet™ iS™10 FTIR spectrometer, shown in Figure 2, was used in the analysis. The samples were placed on a glass side, in most cases using double sided tape to hold them flat. Spectra were collected using the germanium-tip attenuated total reflection (ATR) device. Visual illumination using the bright internal LED of the Nicolet iN5 microscope and on-board camera provided excellent, large area targeting images. This, plus intuitive manual controls, enabled rapid movement to the point of collection. A round aperture was inserted to target the beam. The size of the aperture (1 mm), magnification of the optics (10X) and extra magnification due to the Ge crystal of the ATR (4X) yielded a 25 micron aperture at the sample. The high throughput of the Nicolet iN5 microscope coupled to the Nicolet iS10 spectrometer means that one minute measurements at 8 wavenumber resolution provide excellent signal-to-noise results.

Figure 2: Nicolet iN5 with the Nicolet iS10 FTIR spectrometer.

The target area was centered in the cross hairs and the aperture and Ge-ATR were inserted. A background was collected with the ATR inserted but not in contact with the sample. The preview data collect option in the Thermo Scientific™ OMNIC™ software was enabled, and the stage was slowly raised to bring the sample into contact with the ATR. The appearance of the spectra indicated when contact was sufficient, often at much lower pressures than required to trigger the LED pressure indicators. Excessive pressure could actually penetrate the soft filters, so the preview mode was critical to successful data collection. The data was collected at 8 cm^{-1} resolution and searched against databases using the OMNIC search function.

Results

The first sample analyzed was a filter used to clean milk during production. Figure 3 shows a spectrum of the clean filter, which indicates this is a vinyl acetate material. This is important as many of the residues also exhibited an ester peak (around 1750 cm^{-1}) which is prominent in the filter spectrum.

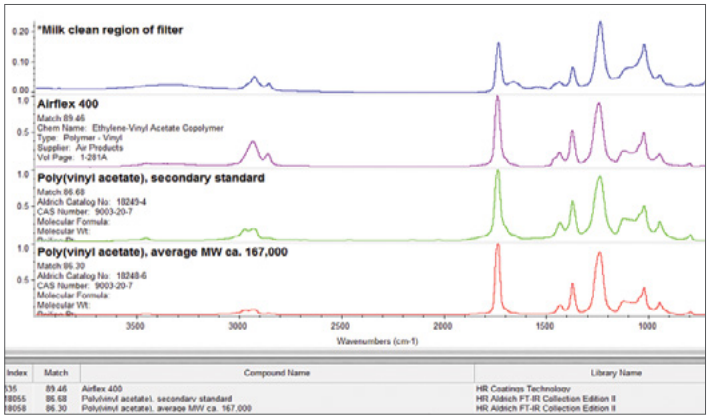

Figure 3: Spectrum of the clean filter, which indicates this is a vinyl acetate material.

Figure 4 is from a region of the filter which was in the flow, but not showing a specific particulate. The entire filter was covered with a white residue, which this spectrum identifies as a fatty acid. This material could be analyzed in two ways, either with the ATR in contact with the filter and then subtracting the clean filter spectrum (Figure 3) or alternately by pressing the ATR down, then lifting it up and analyzing the residue left on the ATR tip itself. The latter is shown in Figure 5 – a significantly cleaner spectrum as no filter background is present and the match result is correspondingly very high.

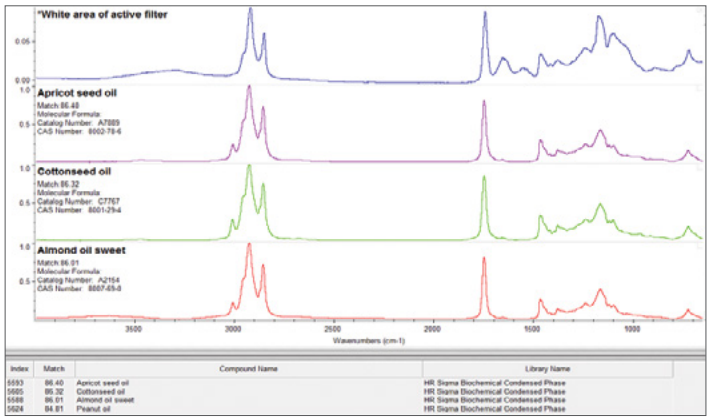

Figure 4: White material on the filter is a fatty acid (triglyceride).

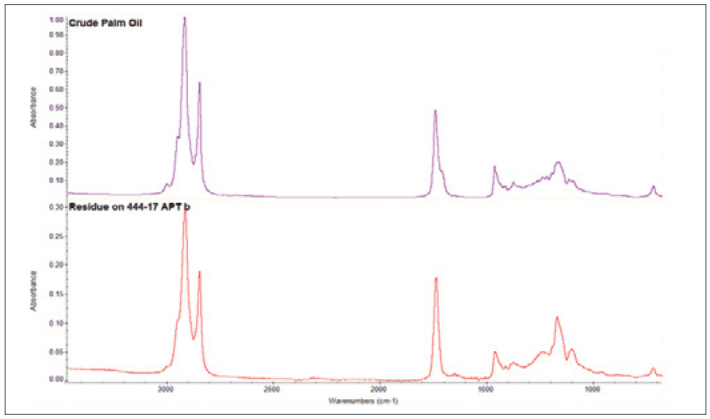

Figure 5: Analysis of the white residue on the ATR tip.

The ATR spectrum of a dark spot on the filter is shown in Figure 6, along with a video image. Subtraction of the spectrum from Figure 4 (filter background plus the white residue) leaves a spectrum showing a strong amide presence (the Amide I and Amide II bands are seen between 1500 and 1700 cm^{-1}) plus other signals over the range from 1000 to 1500 cm^{-1} indicative of sugars and possibly phosphates, all very typical of proteinaceous materials. Milk contains significant amounts of protein such as beta-lactoglobulin, a fat transport protein. These materials can aggregate under the processing conditions, leading to these sticky masses of material on the filter.

Figures 7, 8 and 9 show spectra collected from the inside of a snack food bag. Figures 7 and 8 were acquired in reflectance mode – no ATR used – which was possible because the bag was highly reflective. Again, spectral subtraction was used to remove the background signals due to the bag itself (dominantly polypropylene), allowing the droplet to be identified as an edible oil. The solid brown particle shown in Figure 9 was analyzed using ATR. Subtraction of the spectrum from the greasy coating on the bag yielded a spectrum dominated by a carbohydrate peak. When searched against a common materials library, this spectrum matched to dried potato, a remarkably direct conclusion.

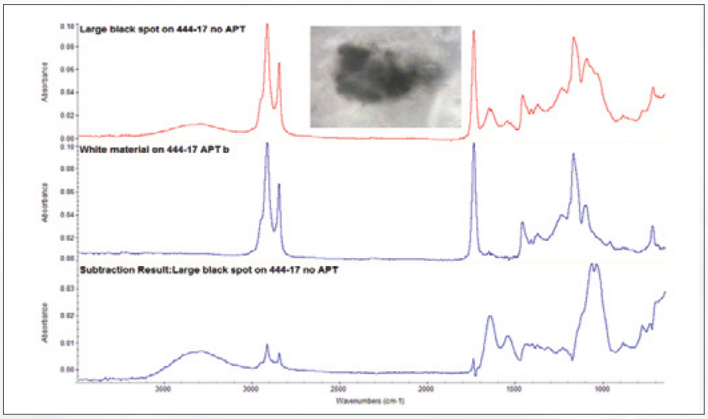

Figure 6: Dark particle contains fatty acid and amide material.

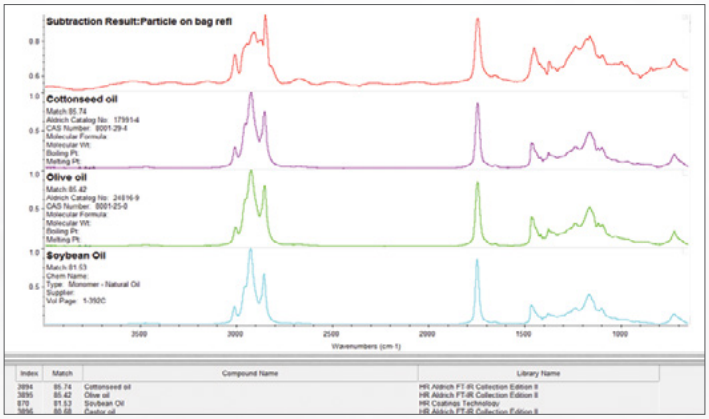

Figure 8: Subtraction results allowing identification of edible oil.

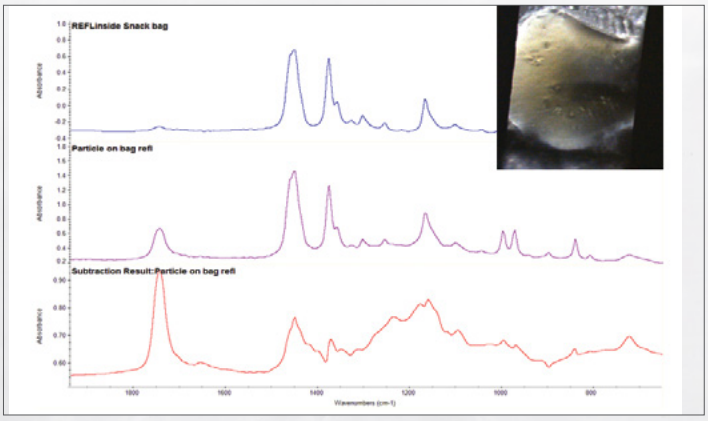

Figure 7: Particles from inside of a snack bag.

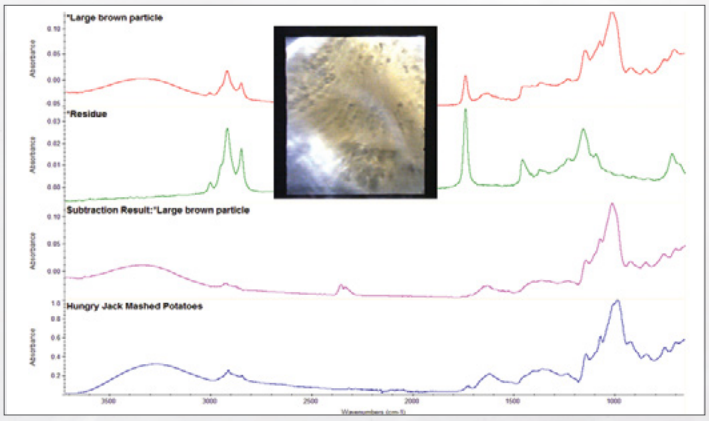

Figure 9: Solid brown particle matched to dried potato.

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Corn meal often exhibits dark granules among the yellow colored majority. An analysis of a yellow and dark granule is shown in Figure 10. The yellow granule matches to starch, which is the majority constituent in corn meal. The darker granule is quite different, appearing to be corn oil. This is probably caused by over processing of a certain part of the sample, and is an example of an acceptable variation in a food product.

The final figure, Figure 11, shows one of the concerns with the analysis of protein material. This is the spectrum of a fly wing, an undesirable contamination. Like most proteins, the spectrum is dominated by the amide bands, seen between 1300 and 1700 cm^{-1}. Since all proteins exhibit these bands, careful analysis is required to determine the origin of the material. In this case, the visual image is a strong clue that the material is a foreign substance. The Nicolet iN5 microscope provides both visual and spectroscopic data to aid in identifications of this type.

Conclusions

Food safety and quality control requires continuous monitoring. The importance of combining both visual data and spectroscopic data is clearly demonstrated. In this example, filters were used as the sample, but extraction of particulates from a bulk sample is also a standard analytical procedure. With a well designed library, the system provides an immediate feedback tool for quality control and diagnostics. The Nicolet iN5 FTIR microscope, combining visual and spectroscopic tools in a cost-effective and simple-to-use package, can drive improvements in quality, regulatory compliance and cost savings.

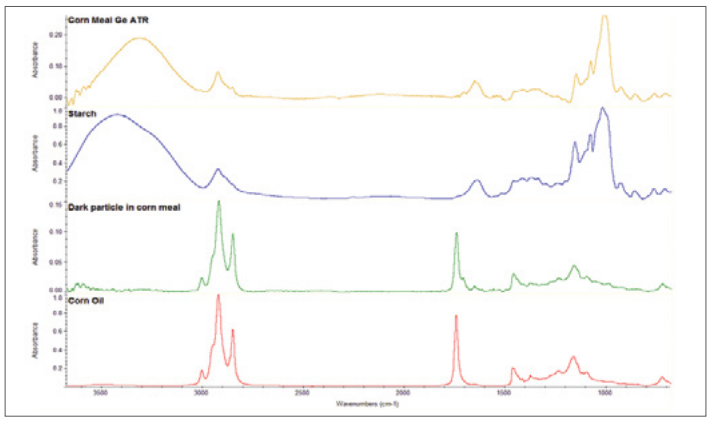

Figure 10: Corn meal analysis.

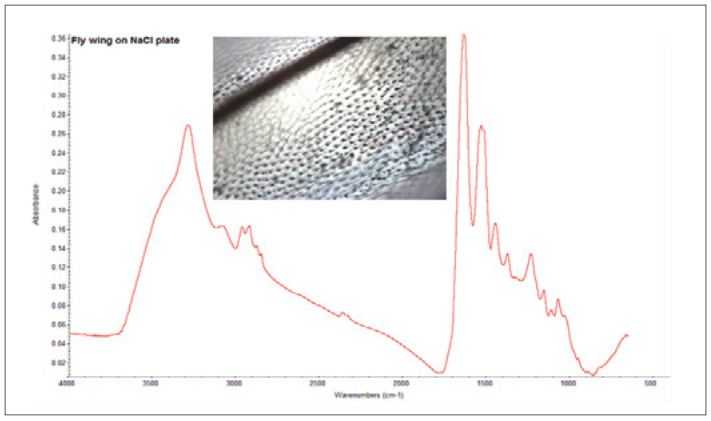

Figure 11: Visual and spectroscopic data aid in identification of fly wing.

Find out more at **www.thermofisher.com/iN5**

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