Application News

No. FTIR-2001

Spectrophotometric Analysis

Visible Defect Analysis in the Consumer Products Industry – Improving Efficiency by Moving Identification Closer to the Problem

■ Introduction

Consumer products manufacturing is a big industry and identification of materials is a consistent need throughout the industry. From incoming material verification to defect or contaminant identification, there is always a need to positively identify materials. Oftentimes, especially in the case of defects and contaminants, the material in question is physically small, but determining the true identity, and likewise the source, is key to solving the problem. The faster the material is identified, the faster the source is found and the faster the line can get back up and running. Identification of the "black spot" is often the gating factor in solving contamination and visible defect issues.

Fortunately, material identification of even small spots can be accomplished using infrared (IR) microscopy. By combining the analytical power of a Fourier transform infrared (FTIR) spectrometer with the precise focus and spatial resolution of a microscope, the chemical identity of samples that are barely visible to the human eye can be identified.

Most visible defects range from 100 – 300 micrometers in size. By focusing and masking the IR beam to the size of the sample, IR microscopes can measure the IR spectrum of a micron-sized area or samples. IR spectra contain information about the chemical bonds present in a sample. By matching the spectrum to a library of known materials, the sample can be easily identified. Measurement times using this technology are quite quick as well. FTIR spectrometers can collect a spectrum in well under a minute for most samples. If sample collection and preparation time is included, results can be obtained in less than 30 minutes in most cases.

So why are visible defects such a concern if they can be easily identified with a relatively short measurement time? The problem is usually where the IR microscope is located. IR microscopes were traditionally expensive and somewhat difficult to use. They often required cryogenic cooling of the detector along with expert alignment to keep them running. Consequently, larger companies purchased IR microscopes for their research and development labs; likewise, smaller companies relied on outside contract labs for microscope analysis. Long-standing backlogs often added to the time required to solve a problem. The analysis may be quick, but sample transportation and sample backlog often lengthen the time until a solution is found. In the end, the root cause of delays in material identification go back to the location of the measurement equipment; moving the measurement technology closer to the problem, and to labs directly affected by the issue, results in faster analysis and consequently substantial cost savings.

■ Instrumentation

Recent advances in IR microscope technology now allow measurements to be made at on-site labs by quality control or process engineers. The IRSpirit™ FTIR spectrophotometer by Shimadzu coupled with the SurveyIR™ FTIR Microscope Accessory by Redwave Technologies, shown in Figure 1, combines a rugged, high-performance design with usability features to offer a system that is simple and durable enough for quality control or production labs. The system can even be transported to remote sites; it installs quickly with no adjustment, facilitating rapid results anywhere it's needed. High-throughput optics allow use with the spectrometer's internal room temperature detector, avoiding the expense and complication of cryogenic liquids commonly used with conventional FTIR microscopes.

Figure 1: SurveyIR FTIR microspectroscopy accessory mounted in IRSpirit FTIR spectrometer's sample compartment.

SurveyIR Microspectroscopy Accessory Advantages

- No alignment
- No maintenance
- Simplified user controls
- View-through, clip-on diamond ATR
- Transmission/Reflection/Oblique illumination modes
- Transmission/Reflection/ATR IR collection modes
- Large, 1900 μm field of view
- 5 MP Digital camera with 2X optical magnification resulting in 0.7 μm/pixel at the sample plane
- Affordable compact design

■ Defect Analysis and Discussion

To show how this analysis works, we'll use an example of a defect found in a plastic component. Visual inspection revealed dark marks throughout a plastic packaging material. The marks were viewed under a stereo microscope and found to be dark, fiber-like contaminants as shown on the left in Figure 2.

Figure 2: (Left) Visible defect found in plastic packaging viewed with a stereo microscope; (Right) Excised fiber rolled flat on a low-E microscope slide and imaged with the SurveyIR

One of these fibers was removed from the plastic using a needle and rolled onto an IR reflective slide for measurement. Under the microscope, the contaminant had a dark blue tint, shown on the right in Figure 2.

The IR spectrum of the contaminant shown in Figure 3 (red) was measured and searched against a spectral library. The blue library match shown in Figure 3 clearly shows that the fiber was made from cotton. Indigo dye was also matched as a secondary component and a reference spectrum is shown in black in Figure 3. The analysis, including sample prep, took less than an hour. With the identity of the material, the plant engineer could now look for the source of the issue.

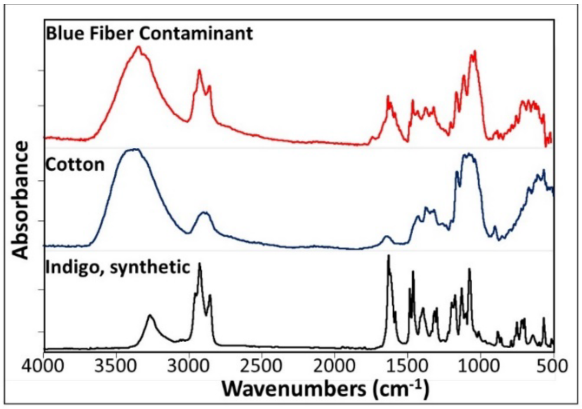

Figure 3: IR spectra of excised blue fiber and corresponding library matches.

Visible defects can also occur in raw materials. Whether these materials are incoming or in use, manufacturers need to identify any visible defect and determine if it will cause a problem in their production. In a recent case, a cosmetics manufacturer discovered a discoloration in the citric acid powder used in one of their products. A sample of the discolored powder was placed on a microscope slide for analysis. The visible image obtained with oblique illumination on the SurveyIR is shown in Figure 4.

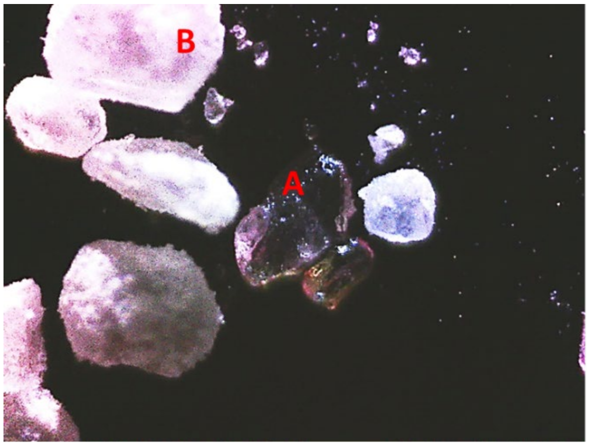

Figure 4: Visible image of contaminated citric acid captured with the SurveyIR microscope using oblique illumination showing both discolored (A) and white (B) crystals.

Oblique illumination provides excellent color representation. Discolored powder is observed in the center of the image compared to the typical white colored citric acid crystals on the outside.

A spectrum of the discolored crystal was measured using the SurveyIR diamond attenuated total reflectance (ATR). ATR is a surface sensitive technique where only the samples directly touching the ATR crystal are measured.

Figure 5 (Red) shows the spectrum of the crystal; features are present from both the citric acid and the contaminant. A comparison to hydraulic fluid is also shown in Figure 5 (Blue) and closely matches the spectral features attributed to the contaminant. A leaking seal on a pump used in the manufacturing process was identified as the source of the hydraulic fluid contamination. Having identified the contaminant, the manufacturer was able to quickly find a compromised seal in a pump and resume manufacturing.

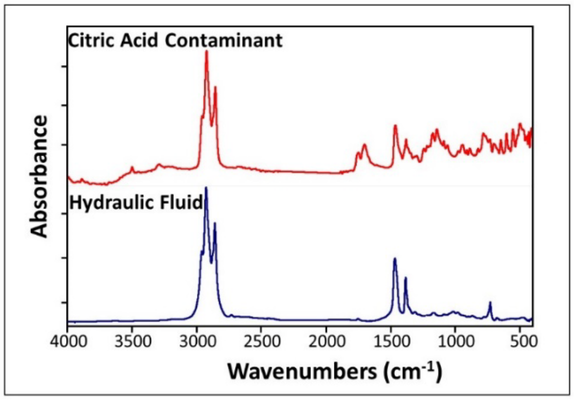

Figure 5: IR spectra collected using the SurveyIR diamond ATR of contaminated citric acid (red) compared to hydraulic fluid (blue) that was identified as the contaminant.

■ Cost Analysis

A simple cost analysis can be used to show the economic advantages of faster sample measurement. We consider the example of a manufacturing production line producing a product leading to $500,000 in yearly revenue, or $1,370 per day. Defects aren't an everyday occurrence, but they may happen six times per year. If the company has an internal R&D laboratory with the capability to analyze these samples, it may take up to three days to get results due to transportation of the samples and laboratory backlog.

In this case, each incident would cost the company approximately $4,000 in lost production or $24,000 per year. Smaller companies may be forced to use an outside laboratory. These labs typically take longer; additionally, each analysis may cost as much as $2,000 per test. In these cases, the company suffers a $5,500 loss in production, plus a cost of $2,000 for each test. The yearly cost may total close to $45,000.

The IRSpirit coupled with SurveyIR system provides a way to reduce these costs by moving the analysis to the quality control or production lab immediately adjacent to the production line. In this case, the analysis can be accomplished in less than half a day; the yearly cost in this case would only be under $4,000 in lost production. Given these numbers, the system would pay for itself in approximately one year compared to using an internal R&D laboratory. For companies using contract labs, the SurveyIR would pay for itself in about 6 months. This calculation only considers savings due to lost production time. By providing higher quality control as well as increased incoming material identification, the total savings in the cost of poor quality can be even higher.

■ Conclusion

Quick identification of contaminants can improve efficiency and reduce costs. New products, such as the compact IRSpirit FTIR spectrophotometer with SurveyIR FTIR microscope, make this analysis accessible to quality control and production laboratories.

■ Acknowledgement

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