SurveyIR™

Using FT-IR Microspectroscopy for the Identification of Contaminants in Paper Products

The global production of pulp and paper products exceeds 400 million tons annually and is growing. With an emphasis on product quality and yield, it is important to identify contamination in paper products and remove the source. Infrared (IR) spectroscopy is ideally suited to characterize unknown solids and the utility of FT-IR microspectroscopy in the microanalysis of pulp and paper contamination was recognized some time ago.[1] With advances in instrumentation, the technique provides better optical performance at reasonable cost bringing FT-IR microspectroscopy within reach of more analysts. This paper will describe the use of the SurveyIR in the FT-IR microanalysis of a small contaminant in a paper product.

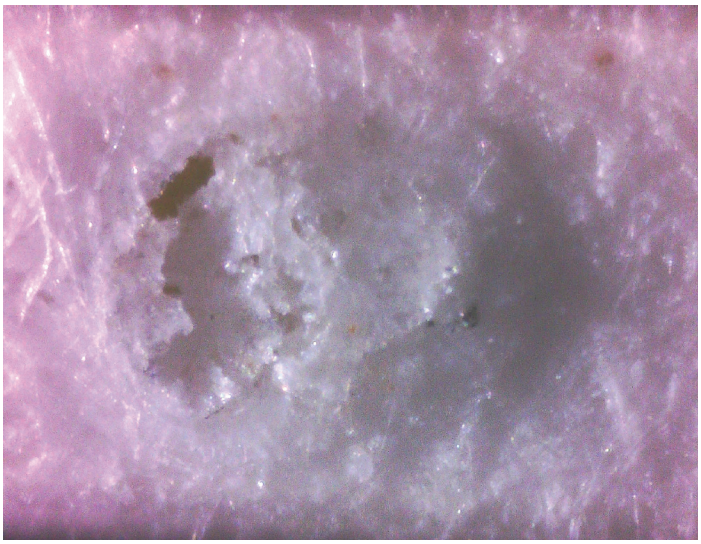

Figure 1: Digital micrograph of contamination on fine paper recorded through the SurveyIR diamond ATR.

Figure 2: Digital micrograph of an excised contaminant recorded on SurveyIR.

A contaminant in fine paper is shown in the digital image in Fig.1, recorded through the diamond ATR prism on SurveyIR using oblique (dark field) illumination. The contaminant appears as a black particle in the upper left section of the image. Discontinuity in the surface of the paper is also noted. It is likely that a larger contamination particle was transferred during the manufacturing process, possibly on the calendar rollers. A similar particle was excised from the paper and placed on a low E glass slide (Fig. 2.) for analysis. The particle was flattened using a stainless steel roller and reflection-absorption spectra were recorded from the low E glass substrate. IR spectra of the particle are shown in Fig. 3. These spectra were measured with a 250 µm aperture using two detector spectrometer-mounted configurations – a narrow-band mercury-cadmium-telluride (MCTA) and deuterated

triglycine sulfate (DTGS). Better signal-to-noise ratio (SNR) is noted in the MCT data but adequate SNR is observed with the DTGS and additional spectral information is obtained due to the lower wavelength response of DTGS. In this case, obtaining this information is crucial in an accurate assessment of the contaminant composition.

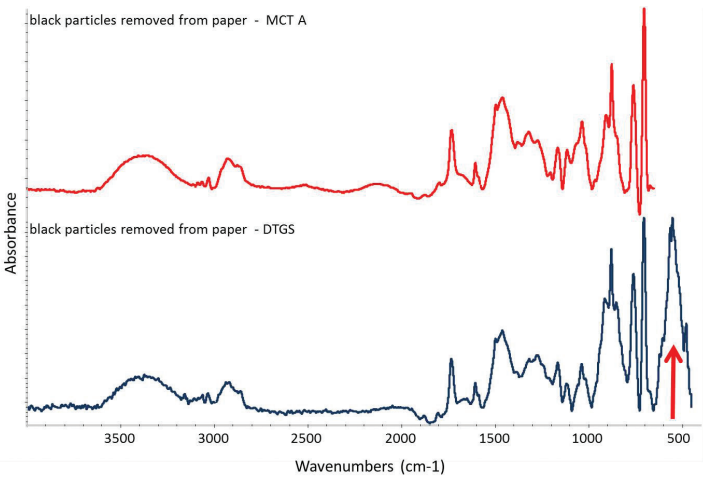

Figure 3: Top: IR spectrum recorded in reflection off low E glass with spectrometer-mounted MCTA detector. Bottom: same sample recorded with spectrometer mounted DTGS.

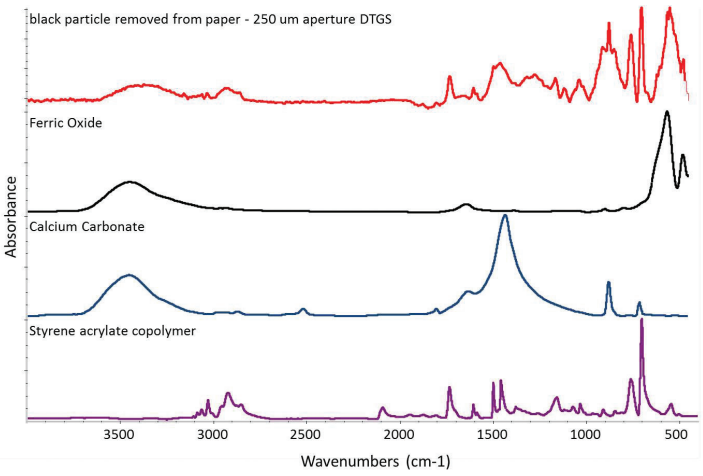

Figure 4: Analysis of contaminant spectrum (top). Iron (II) oxide (middle top), calcium carbonate (middle bottom), and poly (styrene acrylate) (bottom).

The identification of the contaminant particle can be made with the aid of the spectra shown in Fig. 4. The IR spectrum of the contamination particle, recorded with the DTGS detector, is shown in the top of the figure. Very close spectral correspondence is noted with a styrene-acrylate copolymer, shown in the bottom of Fig 4. The contaminant material is largely composed of this copolymer which is very probably poly (styrene n-butyl acrylate). Other significant bands are observed that are not explained by the polymer material. A strong band near 1436 cm^{-1} is best explained by the presence of calcium carbonate. Calcium carbonate is very commonly used both in the polymer and paper industries as a filler or pigment, but can also be present due to high mineral content water used in the paper production. It is difficult to identify the source of the carbonate in this sample. Additional absorption bands are observed near at 560 and 480 cm^{-1} in the contaminant spectrum. These bands are not observed when the photoconductive MCT detector is used because they are below the detector response cutoff. These bands indicate the presence of iron (II) oxide (ferric oxide). A reference spectrum for iron (II) oxide is presented in Fig. 4. The broad absorption near 3400 cm-1 is indicative of hydrated water on either the calcium carbonate or iron (II) oxide.

The source of particle is likely from photocopier toner. Photocopier toner particles are very typically styrene-acrylate polymers filled with carbon black (no observed IR spectrum) and other materials, sometimes iron oxide.[2] This note demonstrates the power of FT-IR microspectroscopy in analyzing complex samples. In this example, a three component mixture was identified from a particle a couple of hundred micrometers in dimension.

References:

1. KM Sweeny, Tappi J, **72**, 171 (1989).

2. J. Brandi, B. James, and S.J. Gutowski, Intl J Doc Exam, **3**(4), 324 (1997).

Micro FTIR Analysis Methods Samples were analyzed "as is" using the Thermo Scientific™ Nicolet™ iS™50 FTIR Spectrometer system.