

Application News

Spectrophotometric Analysis

No.A437

Analysis of Fiber Contaminants by Fourier Transform Infrared Spectrometry (FTIR) and Confocal Laser Scanning Microscopy (CLSM)

Contaminant analysis and fault analysis are essential for conducting product quality management and claim response in a wide range of industries, including food, pharmaceuticals, polymers, electrical equipment, and electronics. To accomplish this, product characteristics such as color and shape are observed, and then the most appropriate analysis method is selected.

Here, we considered the most common type of contaminant as an example, fiber contaminants. Fiber

contaminants include a wide range of substances, such as cotton, acrylic, and polyester, as well as hair of animal origin. Although all of these are easily identified using FTIR, differentiation among those having the same composition is difficult by FTIR. On the other hand, CLSM allows observation of the shape and surface of the sample at the micro level. Here, we introduce examples of analysis of 4 types of fiber contaminants using CLSM and FTIR.

■ Analysis of Fiber Contaminants by FTIR

First, 4 kinds of fiber contaminants (A – D) were selected as samples, and examination was conducted using a stereoscopic microscope (15 × magnification). The observed images are shown in Fig. 1. The differences in color and thickness are clearly visible with the stereoscopic microscope.

Next, we conducted FTIR analysis of the samples to identify them. Fig. 2 shows the instrument used for the identification. Single reflection ATR was used as the measurement technique, which allows an ATR spectrum to be easily obtained by merely pressing the sample against the surface of the prism.

The instrument and analytical conditions used are shown in Table 1, and the ATR spectra obtained with these samples are shown in Fig. 3. With respect to sample A, peaks characteristic of a cellulose system are evident in the vicinities of 3300 cm^{-1} (O-H stretching vibration), 2900 cm^{-1} (third-order C-H stretching vibration), and 1030 cm^{-1} (C-O stretching vibration). Regarding sample B, peaks characteristic of a polyester system are evident in the vicinities of

1710 cm^{-1} (C=O stretching vibration), 1240 cm^{-1} (C-O-C antisymmetric stretching vibration), 1100 cm^{-1} (C-O-C symmetric stretching vibration), and 720 cm^{-1} (C-H, C=O out-of-plane bending vibration) are evident. As for samples C and D, it was evident that these are polyamides (protein) from the existence of characteristic peaks at 3300 cm^{-1} (N-H stretching vibration), 1630 cm^{-1} (C=O stretching vibration), and 1520 cm^{-1} (N-H bending, C-N stretching vibrations). As described above, it was possible to classify the 4 types of fiber contaminants into 3 groups using FTIR analysis.



Fig. 1 Observed Images by Stereoscopic Microscope



Fig. 2 IRAffinity-1 with Attached MIRacle 10 ATR Accessory

Table 1 Instrument and Analytical Conditions

Instrument	: IRAffinity-1, MIRacle10 (Diamond / ZnSe prism)
Resolution	: 4.0 cm^{-1}
Accumulation	: 45
Apodization	: Happ-Genzel
Detector	: DLTGS

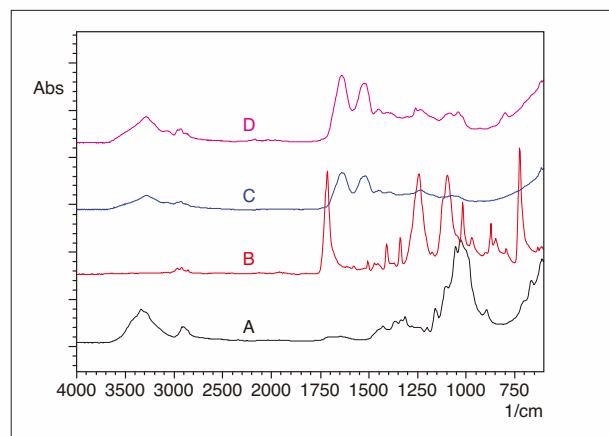


Fig. 3 ATR Spectra by FTIR

■ Analysis of Fiber Contaminants by CLSM

Next we examined fiber contaminants C and D, classified as a single group by FTIR, using the OLS4000 3D measurement confocal laser scanning microscope. A photograph of this instrument is shown in Fig. 4. With the OLS4000, clear images with excellent contrast can be obtained due to sample illumination with the single-wavelength laser light. As a result, 3D observation at high resolution, 3D measurement, and roughness measurement can be conducted.

The instrument and analytical conditions used are shown in Table 2, and the brightness images and 3D images of fiber contaminants C and D are shown in Fig. 5. The cross-sectional diameters were measured from the 3D images. With respect to their shapes, characteristic projections (circled in red) were observed in C, while in D, a dense cuticle was apparent.

From the observation results and the identification results obtained by FTIR, C and D were determined to be polyamide substances, presumably animal hair and human hair, respectively. For comparison, we observed known house cat and human hair samples.

The brightness images and 3D images are shown in Fig. 6. Comparing the results of Fig. 5 and Fig. 6 revealed that the structures C and D closely resembled those of the cat hair and human hair, respectively.

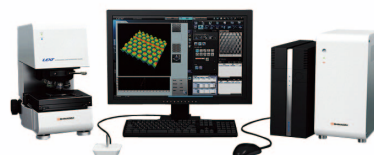


Fig. 4 OLS4000 3D Measurement Laser Microscope

Table 2 Instrument and Analytical Conditions

Instrument	: OLS4000
Laser	: 405 nm
Objective lens	: 100 × (NA 0.95)
Field of view	: 128 μm × 96 μm

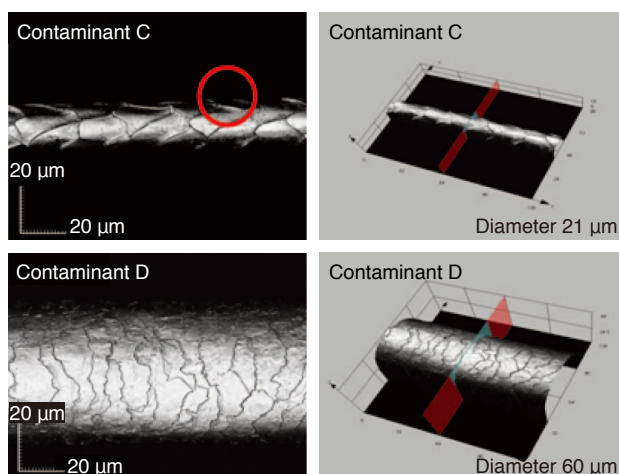


Fig. 5 Brightness Images (left) and 3D Images (right) by CLSM

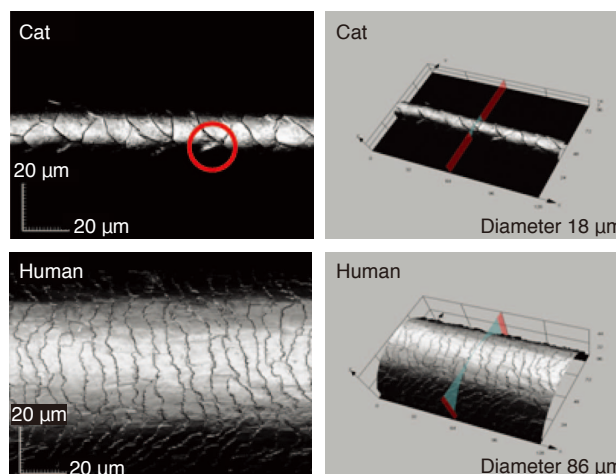


Fig. 6 Brightness Images (left) and 3D Images (right) by CLSM

■ Conclusion

We analyzed 4 kinds of fiber contaminants using FTIR and CLSM. Qualitative analysis of each of the samples was possible by FTIR, and using CLSM, we were able to differentiate between samples having the same composition.

Here, we were able to conduct measurement by single reflection ATR using FTIR because the samples were large enough to be clearly visible. Micro-level samples

in the range of 10-100 μm can be effectively analyzed by infrared microscopy.

In contaminant analysis and fault analysis, observation of color and shape both visually and using stereoscopic microscopy is important. Following such observation, more detailed information can be obtained using multiple analytical instruments, as presented in this example.



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