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Application News

Infrared Microscope
Infrared Raman Microscope

Analyzing Microscopic Contaminants Embedded in Recycled Plastic

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User Benefits

- Microscopic contaminants embedded in recycled plastic can be analyzed with an infrared microscope using a simple method.
- The type, number, and shape of contaminants in recycled plastic can be easily determined.
- Data can be obtained quickly through the following steps: use a thermal press to form the recycled plastic into a film; perform high-speed mapping analysis by transmission spectroscopy; and then analyze the data with a particle analysis program.

■ Introduction

Reusing plastic waste is key to achieving a carbon-neutral society, which is why targets have been established for levels of recycled plastic in plastic products. But quality control of recycled plastics is essential for maintaining the functional properties of plastic products since improper sorting, transporting, and processing can all lead to contamination of recycled plastic. Determining the type and quantity of contaminants in recycled plastic is important since they can have a significant negative impact on product performance in certain applications.

Shimadzu's infrared microscopes use high-speed mapping analysis to quickly find and characterize contaminants dispersed in recycled plastic, and Shimadzu's particulate analysis program also provides a convenient and simple tool for determining the morphology and numbers of contaminants.

This Application News describes using this equipment and tools to analyze contaminants in recycled plastic material that was formed into a film.

■ High-Speed Mapping

Outline of the High-Speed Mapping Feature

Fig. 1 shows a flowchart and graphical representation of the high-speed mapping feature. A single scan is performed of each area of the sample, and if a peak is detected in that area, a preset number of scans is then repeated in that area. If no peak is detected in the first scan, the spectrum from this scan is saved, and the infrared microscope then moves to the next location. The parameters that determine peak detection are configured in the high-speed mapping settings and include noise level, threshold value, and exclusion ranges. Up to 10 different configurations of these high-speed mapping settings can be saved for future use.

High-speed mapping analysis is when the targets of interest are spread over a wide sample area. Two examples of this are contamination in raw materials and microplastics captured on a filter. In both these cases, high-speed mapping offers the ability to (1) quickly analyze targets of interest spread over a wide area while (2) recording infrared spectra for target characterization.

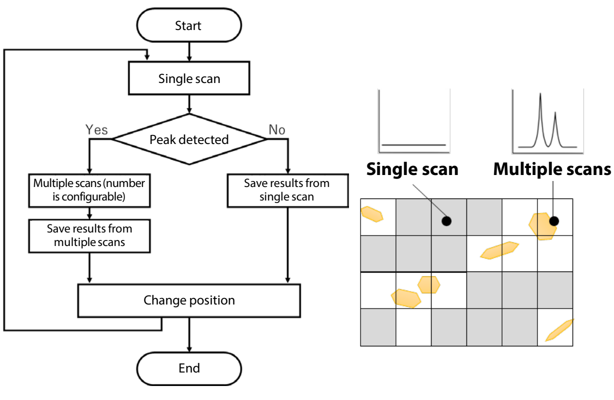

Fig. 1 Flowchart (Left) and Graphical Representation (Right) of High-Speed Mapping Feature

High-Speed Mapping Analysis of Recycled Plastic

For recycled plastic with a relatively simple infrared spectrum, contaminants embedded in the plastic can be analyzed without removing them from the plastic. This is achieved by configuring wavenumber ranges (exclusion ranges) that exclude the peaks of the recycled plastic in the settings of the high-speed mapping software. In this example, a thermal press was used to form recycled plastic composed of PE and PP into a plastic film approximately 100 µm thick. The high-speed mapping settings were then configured to detect peaks in wavenumber regions that contain hydroxyl groups and carbonyl groups that are not normally found in PE or PP. Table 1 shows the analysis conditions in detail. Background readings were obtained from an area of the film with no contaminants.

Table 1 Analysis Conditions

Equipment:	IRTracer™-100, AIMsight™
Software:	AMsolution, high-speed mapping software, particle analysis program
Optical System:	Transmission
Resolution:	8 cm^{-1}
Number of Scans:	50
Apodization Function:	SqrTriangle
Mirror Speed:	30 mm/sec
Aperture Size:	30 µm × 30 µm
Step Size:	30 µm
Mapping Range:	2,370 µm × 1,470 µm
Detector:	T2SL
High-Speed Mapping Settings	
Noise Level:	0.01
Threshold Value:	0.15
Excluded Ranges:	3,200-2,000 cm^{-1}, 1,700-700 cm^{-1}

■ Results

Infrared Spectra of Contaminants

Fig. 2 shows infrared spectra obtained from the high-speed mapping analysis. The black line labeled Skip is the infrared spectrum at a position in the sample where no peaks were detected and only a single scan was performed. The infrared absorption at this position is almost identical to the background spectral measurement and contains no significant peaks. Looking at the positions on the sample where multiple scans were performed reveals two types of infrared spectra. Fig. 2 shows two representative examples of these spectra in blue and green. A spectral search in wavenumber ranges that excluded 3,200 to 2,700 cm^{-1}, where there is strong infrared absorption by the base material (PE and PP), identified the green line as cellulose and the blue line as polybutyl methacrylate (PBMA).

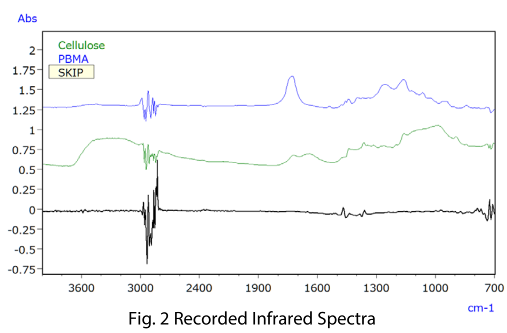

Fig. 2 Recorded Infrared Spectra

Particle Analysis of Contaminants

Shimadzu's infrared microscopes are controlled by AMsolution software, which comes with an optional particle analysis program that provides a simple method for determining the number, size, and shape of contaminants in a specific area of interest. In this instance, the infrared spectra of the cellulose and PMBA detected in the plastic film were loaded into the particle analysis program, which was then configured to detect regions with a spectral similarity of 600 or above and particle sizes in the range of 100 to 10,000 µm^2.

Fig. 3 shows the positions of contaminants in a piece of the plastic film. They are present in a variety of shapes and sizes over a wide area of the film.

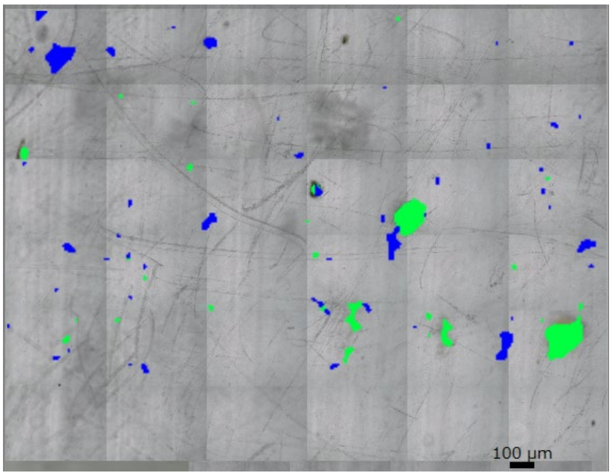

Fig. 3 Image Showing Contaminants in Recycled Plastic Film
(Green: cellulose, Blue: PBMA)

The bar chart in Fig. 4 shows the number of individual contaminants detected by the particle analysis program. The program detected 74 contaminants within the analyzed area (2,370 × 1,470 mm), which contained about twice as many individual PBMA contaminants as cellulose contaminants.

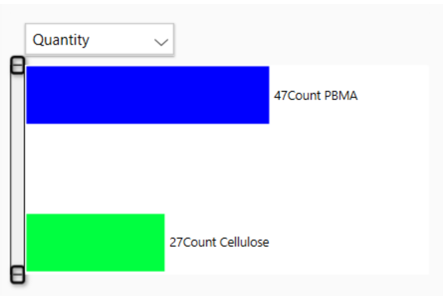

Fig. 4 Numbers of Contaminants in Recycled Plastic Film

Fig. 5 shows a histogram of contaminant sizes based on short diameter (µm), and Fig. 6 shows the same information based on long diameter (µm). Based on short diameter, the proportion of cellulose and PBMA contaminants in each size category was similar to the proportion of cellulose and PBMA contaminants detected overall. With the long diameter, the size range of 20 to 30 µm contained an almost equal proportion of cellulose and PMDA contaminants. This shows that the smaller cellulose contaminants were fiber-shaped.

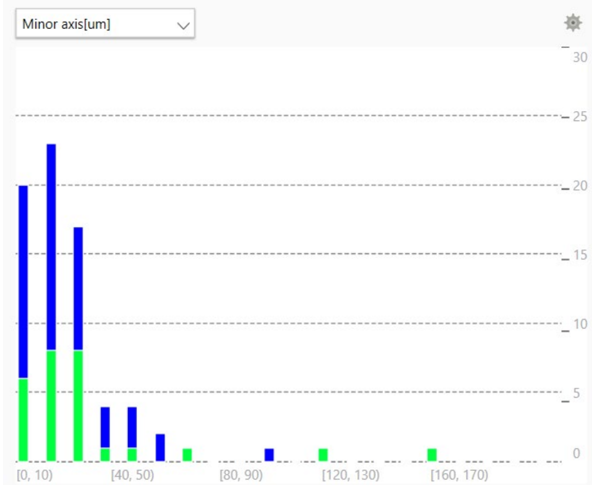

Fig. 5 Histogram of Contaminants by Short Diameter (µm)

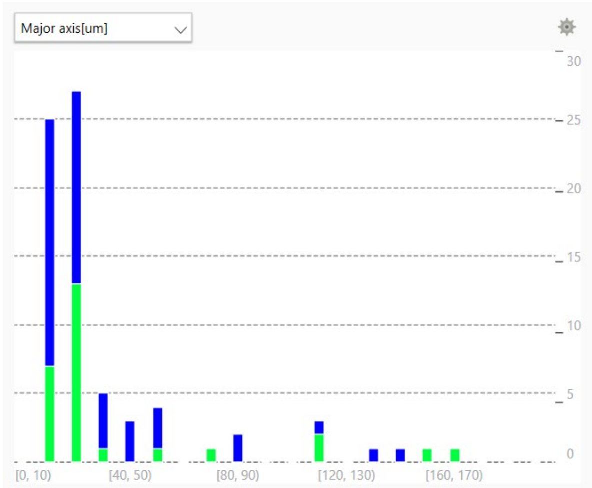

Fig. 6 Histogram of Contaminants by Long Diameter (µm)

■ Conclusion

This Application News describes a simple and rapid method for analyzing and characterizing contaminants in recycled plastic using high-speed mapping analysis with an infrared microscope and data analysis with a particle analysis program. Areas of recycled plastic with no contaminants were scanned once, whereas areas with contaminants were scanned multiple times (50). This allowed a wide area of recycled plastic to be analyzed quickly with minimal noise in the recorded infrared spectra, reducing the analysis time to around just one-fifteenth of the time needed if every part of the recycled plastic was scanned 50 times. Also, using a thermal press to form recycled plastic pellets into a film enabled the efficient characterization of large numbers of contaminants by transmission spectroscopy and the ability to calculate their numbers in the pellets.

<Related Applications>

1. High-Speed Measurement of Microplastics Smaller than 100 µm Collected on a Filter and Efficient Analysis—Using a High-Speed Mapping Program and Particle Analysis Program—
Application News No. 01-00994-EN

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