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Application News

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Spectrophotometric Analysis

Analysis of Microplastics by iSpect™ DIA-10 Dynamic Particle Image Analysis System and AIM-9000 Infrared Microscope

Fine plastics with sizes on the order of several μm to several mm are called microplastics. Microplastics have become a global issue in recent years as a marine environmental problem that not only has adverse effects on coastal and marine ecosystems, but potentially may also affect human health. In order to protect the global environment, an early response is necessary. Various types of devices are used in identifying the sources of microplastics and studying improvement measures.

A dynamic particle image analysis system is suitable for analysis of the shape and particle count concentration (particles/mL) of microplastics dispersed in solutions. Although analysis of individual microplastic particles is also possible by stereomicroscope, the burden on the operator is large and analysis efficiency is poor. A dynamic particle image analysis system can automatically detect particles with sizes of 5 to 100 μm dispersed in a solution and analyze their shape and particle count concentration in a short time.

Infrared microscope, which excels in analysis of organic compounds, is suitable for qualitative analysis of microplastics with sizes of 100 μm or less that can be captured with filter paper.

Here, we introduce an example of analysis of the shape and particle count concentration of particles contained in environmental water and their qualitative analysis by using a dynamic particle image analysis system and an infrared microscope.

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iSpect DIA-10 Dynamic Particle Image Analysis System

The iSpect DIA-10 shown in Fig. 1 measures particles by the microcell method, in which image acquisition efficiency is enhanced by passing the particles through a narrow imaging area. Because fewer particles pass outside the imaging area (outside the imaging area to the right or left sides) and blurring in the front and back directions is small in comparison with the conventional method, and virtually all particles are captured, the microcell method enables highly reliable particle detection. Moreover, because samples as small as 50 μL can be measured, this method also supports measurement of scarce samples. As shown in Fig. 2, measurements can be conducted by setting the tip of a general-purpose pipette in the top of the instrument.

Fig. 1 iSpect™ DIA-10 Dynamic Particle Image Analysis System

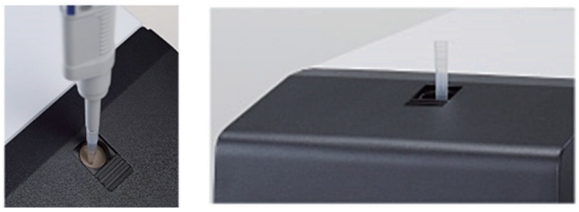

Fig. 2 Sample Setting

AIM-9000 Infrared Microscope

The infrared microscope system shown in Fig. 3 makes it possible to acquire information on microscopic areas with high sensitivity by using the aperture to narrow the infrared light beam to the designated size. Visual observation of microplastics on filter paper is also easy by using the digital zooming function of Shimadzu proprietary wide-field camera* and microscope camera, which enables zooming to a magnification of 330×.

* The wide-field camera is available as an option.

Fig. 3 IRTracer™-100 Fourier Transform Infrared Spectrophotometer (Left) and AIM-9000 Infrared Microscope (Right)

Analysis of Shape and Particle Count Concentration of Particles in Environmental Water

Environmental water containing microplastics was used as the sample, and the shape and particle count concentration of the particles contained in the sample were analyzed with an iSpect DIA-10. Table 1 shows the measurement conditions, and Fig. 4 shows a portion of the acquired particle images.

Table 1 Measurement Conditions

Instrument	: iSpect DIA-10
Frame rate	: 8 fps
Analysis flow rate	: 0.1 mL/min
Total analyte	: 150 μL

From Fig. 4, the shapes of particles with sizes of 100 μm and less have be captured clearly, and various shapes, such as rod-like and fibrous shapes, can be confirmed.

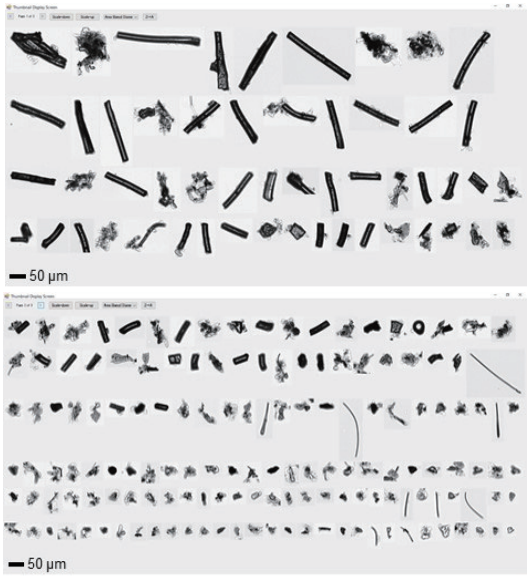

Fig. 4 Particle Images

Fig. 5 and Fig. 6 show the scattergram (scatter diagram) and histogram (frequency distribution graph), respectively (range shown on horizontal axis: 10 to 100 µm). Scattergrams and histograms can be prepared by selecting two desired measurement items (e.g., maximum length, aspect ratio, circularity). The results showed that the particle count concentration was 5,309 particles/mL. The average size was 24.315 µm, and the largest number of particles was in the size range of 10 to 30 µm, as shown by the red box in Fig. 6.

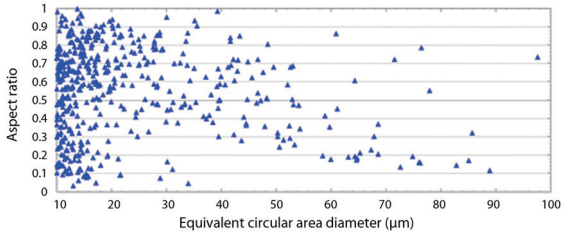

Fig. 5 Scattergram (Scatter Diagram)

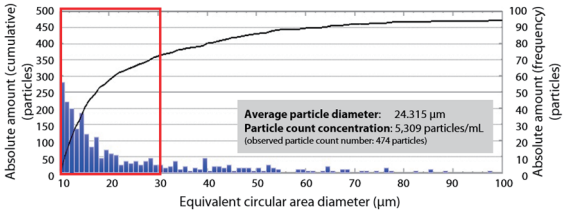

Fig. 6 Histogram (Frequency Distribution Graph)

Qualitative Analysis of Microplastics

After the measurement by iSpect DIA-10, the particles contained in the sample were captured with polytetrafluoroethylene (PTFE) filter paper and a mapping analysis was done with an AIM-9000. Table 2 and Fig. 7 show the measurement conditions and a visual observation image, respectively.

Table 2 Measurement Conditions

Instruments	: IRTracer-100, AIM-9000
Resolution	: 8 cm^{-1}
Accumulation	: 5 times
Apodization function	: Sqr-Triangle
Aperture size	: 20 µm × 20 µm
Mapping range	: 460 µm × 1,780 µm
Detector	: MCT

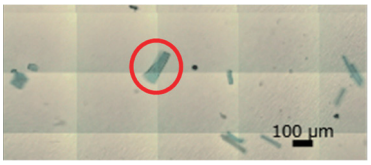

Fig. 7 Visual Observation Image

As a result of the qualitative analysis of the infrared spectrum (Fig. 8) of the area in the red circle in Fig. 7, this particle was identified as polypropylene (PP).

Next, Fig. 9 shows a chemical image prepared using the corrected area value (area value of peak above the baseline) of the characteristic peak of PP in the range of 1,400 to 1,339 cm^{-1} (CH_3 bending vibration). The color red shows areas with a large content of the component PP, and blue shows areas with a small content. This result clarified the fact that all of the rod-like microplastics that can be observed in the visual observation image are PP.

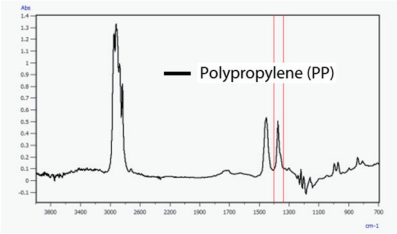

Fig. 8 Infrared Spectrum of Rod-like Microplastics (The red box shows the peak used in preparing the chemical image in Fig. 9.)

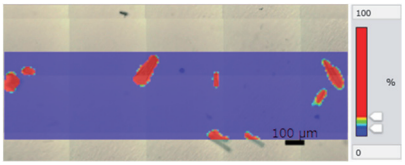

Fig. 9 Distribution of PP (Corrected Area Value of 1,400-1,339 cm^{-1} Peak)

Conclusion

By using the iSpect DIA-10 dynamic particle image analysis system, the shapes of the large number of particles contained in an environmental water sample could be observed efficiently, and statistical values for the size and shape and information on the particle count concentration could be obtained. The AIM-9000 infrared microscope enabled quick qualitative analysis of the composition of the microplastics captured by visual observation images, and its distribution could be shown clearly by using a chemical image.
These systems realized a hybrid analysis of the shape and particle count concentration of microplastics and identification of their composition, thus providing a variety of knowledge.

<Acknowledgments>
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