Application News

AIRsight™ Infrared/Raman Microscope

Analysis of Microplastics Using AIRsight Infrared/Raman Microscope

Kazumi Kawahara

User Benefits

- By using AIRsight, infrared and Raman measurements can be made on the same stage without moving the sample.
- Sample length can be measured from images acquired with a wide-field camera or an objective lens for infrared or Raman measurements.
- Enables the material of microplastics in the environment to be accurately determined.

■ Introduction

Microplastic pollution of rivers and oceans is spreading globally, and there are concerns about its impact on living organisms. In recent years, monitoring surveys and research have been conducted to gain scientific knowledge about the distribution of microplastics in many countries worldwide. When exposed to UV light, rain, wind, and physical friction that makes it brittle, the plastic released into the environment breaks up into even smaller microplastics. (The microplastics here are called secondary microplastics.). Generally, microplastics are evaluated by observing their appearance, measuring their number and size, and qualifying their materials. Among these items, qualitative measurement of the material is one of the most important items for identifying the origin of the microplastic. However, the size of microplastics to be evaluated is becoming smaller year by year, requiring the selection of appropriate analytical instruments. A size-specific analysis method for microplastics is shown in Figure 1. Micro-Raman spectroscopy makes it possible to analyze smaller particles than micro-infrared spectroscopy and is easier to analyze than pyrolysis-gas chromatography-mass spectrometry. The AIRsight Infrared/Raman microscope is a new type of microscope that incorporates a Raman unit into an infrared microscope, enabling both Raman and infrared analysis to be performed on a single instrument, analyses that previously required separate instruments. This article introduces an example of evaluating microplastics in the environment using AIRsight.

Fig. 2 Appearance of IRXross™(left) and AIRsight™(right)

■ Microplastics Used for Measurement

Microplastics in water were filtered using paper made of PTFE (polytetrafluoroethylene) and collected on the filter paper. (PTFE has no infrared absorption except around 1200 cm^{-1}, so microplastics can be measured by the transmission method with the filter intact.) The microplastics collected on the filter paper were placed on the stage of the AIRsight Infrared/Raman microscope for infrared and Raman measurements. Figure 3 shows images of microplastics on filter paper taken with the infrared and the Raman objective lenses. In this article, three different sizes (a), (b), and (c) of microplastics were measured.

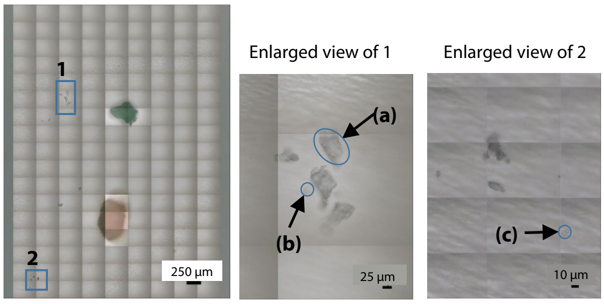

Fig. 3 Microplastic Image Taken with Objective Lens

■ Qualitative Analysis by Microscopic Infrared Spectroscopy

Microplastics (a) collected on filter paper were measured by the transmission method with an infrared microscope. The measurement conditions are shown in Table 1. In addition, Figure 4 shows search results using Shimadzu's own UV-Damaged Plastic Library.

Table 1 Measurement Conditions

Instruments:	IRXross™, AIRsight
Infrared Spectrometry	
Resolution:	8 cm^{-1}
Accumulation:	30
Apodization Function:	Happ-Genzel
Aperture Size:	25 µm
Detector:	T2SL

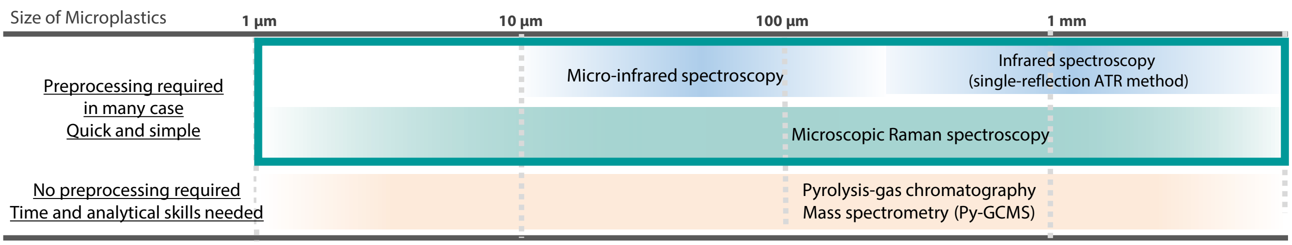

Fig. 1 Methods for Analyzing Microplastics by Size

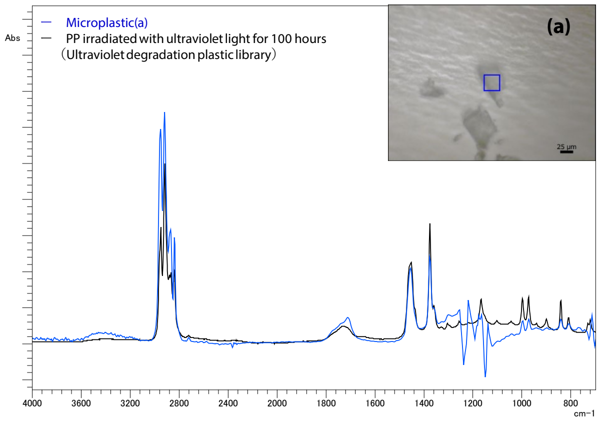

Fig. 4 Infrared Spectrum of Microplastics (a) on Filter Paper

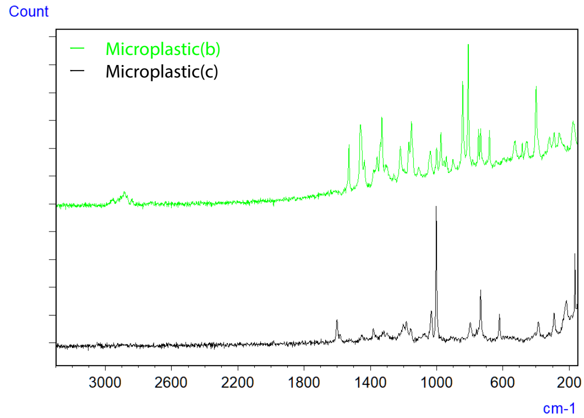

Fig. 6 Raman Spectra of Microplastics (b) and (c) on Filter Paper

Microplastic (a) was found to have a spectrum similar to that of PP (polypropylene) irradiated with UV light for 100 hours. The noise around 1,200 cm^{-1} is due to absorption by PTFE, the material of the filter paper.

Although the search results are not shown, it was determined from the Raman spectra that microplastic (b) was PE (polyethylene) and (c) was PS (polystyrene).

■ Qualitative Analysis by Micro-Raman Spectroscopy

Micro-Raman spectroscopy was used to measure microplastics of smaller sizes, which are difficult to measure by infrared microspectroscopy. The images of microplastics (b) and (c) taken by the objective lens are shown in Fig. 5, the measurement conditions are shown in Table 2, and the resulting Raman spectra are shown in Fig. 6. In Raman spectroscopy, measurements are generally made at an excitation wavelength of 532 nm, where Raman scattering is strong. While the peak intensity is sufficient, it is difficult to obtain good data in the case of fluorescent samples because the baseline rises due to the fluorescence. Since many microplastics degraded by UV light are known to emit fluorescence at an excitation wavelength of 532 nm[1], measurements were performed at an excitation wavelength of 785 nm for this article. Compared to the excitation wavelength of 532 nm, measurement at 785 nm has a shorter wavenumber range due to the detector characteristics. However, it has the advantage of reducing fluorescence.

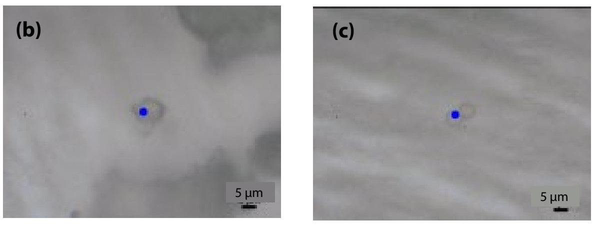

Fig. 5 Images of Microplastics (b) and (c) Taken with the Objective Lens

Table 2 Measurement Conditions

Instruments:	IRXross™, AIRsight
Raman Spectrometry	
Accumulation:	40
Exposure Time:	5.0 sec
Objective Lens:	100x
Excitation Wavelength:	785 nm
Detector:	CCD

■ Length Measurement Function

Using the images acquired here, we introduce a new function of AIRsight control software AMsolution, the length measurement function. You can measure the length of an image captured with a wide-field camera or objective lens by setting its start and end points. The operation screen is shown in Figure 7. This feature provides size information for microplastics as well as material information. The major diameters of microplastics (a), (b), and (c) were 97 µm, 10 µm, and 5 µm, respectively.

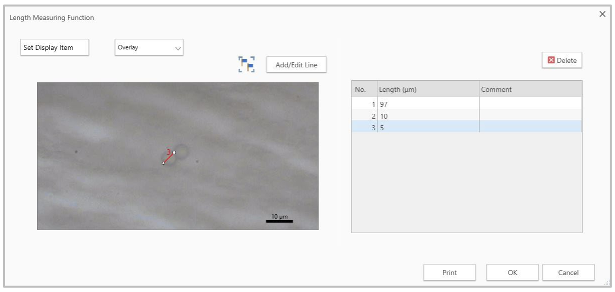

Fig. 7 Length Measurement Operation Screen

■ Conclusion

In this article, microplastics of various sizes were measured and qualified using the AIRsight Infrared/Raman microscope. Micro-infrared spectroscopy allows the measurement of microplastics down to about 10 µm in size. However, by using this in combination with micro-Raman spectroscopy, it is possible to measure microscopic samples below 10 µm, which are difficult to measure by micro-infrared spectroscopy alone. In addition, by using the length measurement function, it is possible to not only identify the microplastic material but also obtain the size information.

Reference Documentation
1) Zenjiro Osawa: "Principles of Chemiluminescence Method and Applications to Polymer Degradation," Material Life, Vol. 3, No. 1, pp. 32 -39 (1991)

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