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Application News

AIRsight™ Infrared/Raman Microscope

Analysis of Microplastics Using AIRsight Infrared/Raman Microscope

K, 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.
- It can accurately determine the material of microplastics in the environment.

■ Introduction

Pollution of rivers and oceans by microplastics is spreading on a global scale, and there are concerns about its impact on living organisms. In recent years, monitoring surveys and research have been actively conducted to obtain scientific knowledge about the distribution of microplastics in around the world. When exposed to UV rays, rain and wind, and brittle by physical friction, the plastic released into the environment becomes even smaller, becoming microplastics.(The microplastics here are called secondary microplastics.). Generally, evaluation items for microplastics include observation of their appearance, measurement of their number and size, and qualitative of materials. Among these evaluation items, Qualitative of material is one of the most important items for identifying the origin of microplastics, but the size of microplastics to be evaluated is getting 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 is possible to analyze smaller particles than micro-infrared spectroscopy and is easier to analyze than pyrolysis-gas chromatography-mass spectrometry. AIRsight Infrared/Raman microscope is a new type of microscope that incorporates a Raman unit into an infrared microscope, making it possible to perform both Raman and infrared analysis on a single instrument, which was previously required for each instruments had been required until now. This article introduces an example of evaluating microplastics in the environment using AIRsight.

Fig. 2 Appearance of AIRsight™

■ Microplastics used for measurement

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

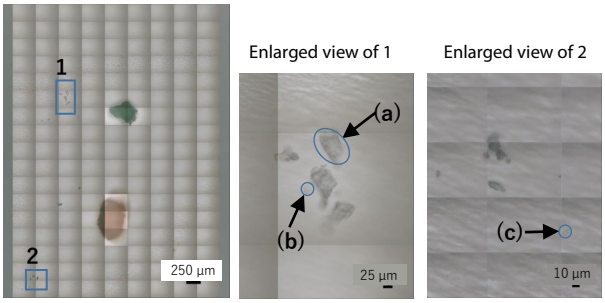

Fig. 3 Microplastic Image taken with Objective Lens

■ 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-Dameged 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

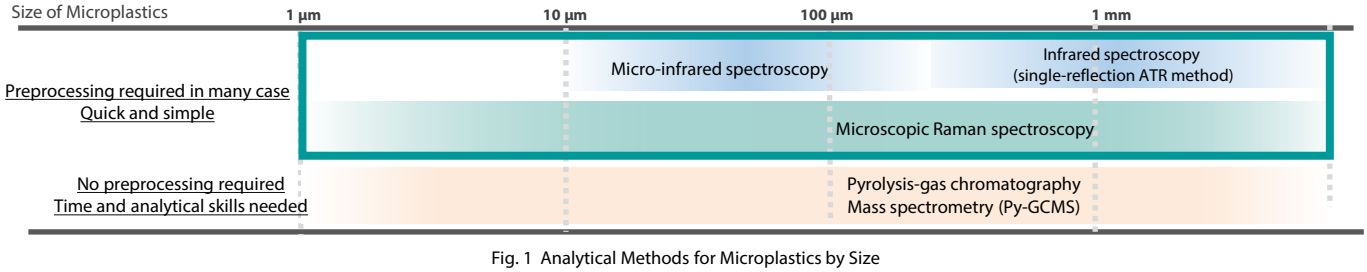

Fig. 1 Analytical Methods for Microplastics by Size

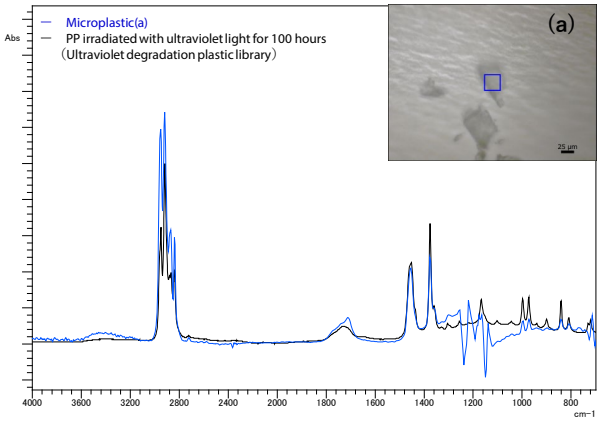

Fig. 4 Infrared Spectrum of Microplastics (a) on filter paper

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

■ 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 intense, but while peak intensity is sufficiently obtained, it is difficult to obtain good data in the case of fluorescent samples because the baseline rises due to the influence of fluorescence. Since many of microplastics degraded by UV rays are known to emit fluorescence at an excitation wavelength of 532 nm[1], in this article, we performed measurements at an excitation wavelength of 785 nm. Compared to the excitation wavelength of 532nm, the measurement at 785nm has a shorter wavenumber range due to the characteristics of the detector. However, it has the advantage of reducing to fluorescence.

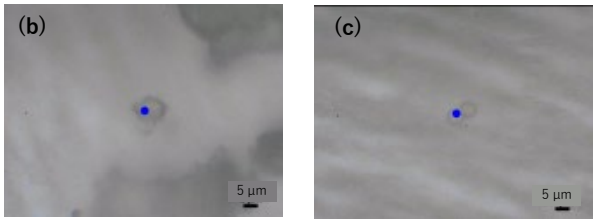

Fig. 5 Images of Microplastics (b) and (c) taken with the Objective Lens

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

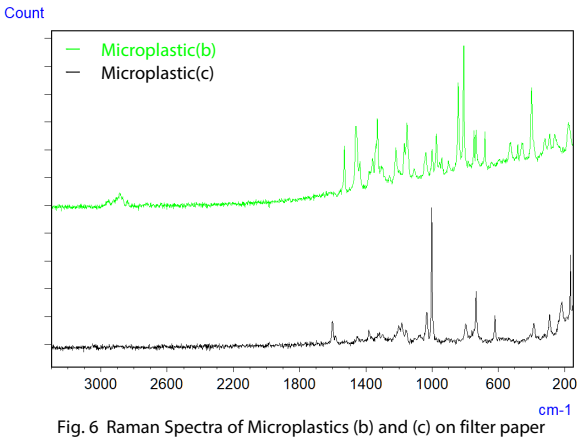

Fig. 6 Raman Spectra of Microplastics (b) and (c) on filter paper

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

■ 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.

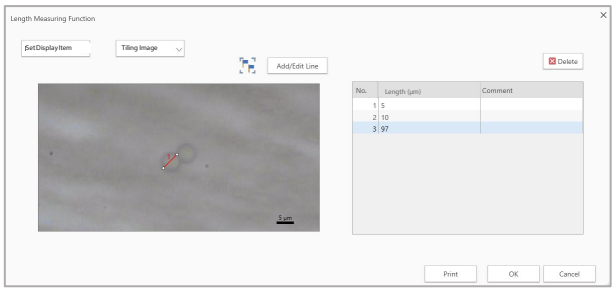

Fig. 7 Length Measurement Operation Screen

■ Conclusion

In this article, microplastics of various sizes were measured and qualified using Infrared/Raman microscope, AIRsight. Micro-infrared spectroscopy afford to measure microplastics up to about 10 µm, however, by using micro-Raman spectroscopy in combination, it is possible to measure even microscopic samples below 10 µm, which is difficult to measure by micro-infrared spectroscopy. In addition, by using the length measurement function for these measurement results, not only the material of the microplastics can be identified, but also the size information can be obtained.

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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