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Application News

IRSpirit™ Fourier Transform Infrared Spectrophotometer

Sample Preparation Method and FTIR Analysis Method for Microplastics Sampled from Rivers

R. Fuji

User Benefits

- Simple degradation analysis of microplastics is possible by using the Plastic Analyzer plastic analysis system.
- In analysis of microplastics, adequate sample preparation enhances the accuracy of the subsequent analysis.

■ Introduction

Microplastics are fine plastic particles with a size of 5 mm or smaller. Because pollution of rivers and oceans by microplastics is spreading at the global scale, monitoring surveys have been conducted in recent years to obtain scientific knowledge concerning the distribution of microplastics in each of the world's countries and various other issues. Standardization of monitoring survey methods is also being promoted so that the data from each country can be compared. Analysis of microplastics is generally carried out in the order of collection of samples, sample preparation (removal of contaminants by hydrogen peroxide, classification of the microplastics by gravity separation), and evaluation by analytical instruments. Adequate sample preparation is indispensable for high accuracy in the subsequent analysis step.

This article introduces the condition of microplastic sampling, a sample preparation method, and an analysis method using the Shimadzu Fourier transform infrared spectrophotometer (FTIR) shown in Fig. 1.

Fig. 1 IRSpirit™ Fourier Transform Infrared Spectrophotometer

■ Condition of Microplastic Sampling

Microplastics were sampled from rivers in Kyoto Prefecture using an Albatross microplastic sampling device manufactured by Pirika, Inc. Albatross was developed to solve the problems of cost (high boat rental cost) and constraints (difficulty of surveying narrow waterways and rivers with slow flows), which had been an obstacle to surveys, and can be used easily by anyone.

Fig. 2 shows the appearance of the Albatross sampling device, Fig. 3(a) and (b) show the condition of sampling, and Fig. 4 shows a collected sample. Sampling was carried out by submerging Albatross in the water for 3 minutes.

Fig. 2 Albatross Microplastic Sampling Device

Fig. 3(a) Condition of Sampling in River A

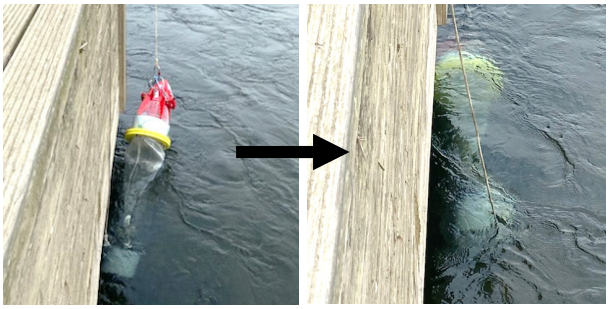

Fig. 3(b) Condition of Sampling in River B

Fig. 4 Collected Sample

■ Microplastic Sample Preparation Method

First, the collected sample was passed through sieves with mesh sizes of 2 mm and 0.1 mm, and 30 % hydrogen peroxide (H_2O_2) was added to the particles captured on the 0.1 mm sieve to dissolve contaminants. Next, the H_2O_2 was removed and the sample was washed with pure water, 5.3 mol/l of aqueous sodium iodide (NaI) was added, and gravity separation was conducted. Fig. 5 shows the workflow of the above sample preparation procedure, and Fig. 6 shows some of the captured microplastics.

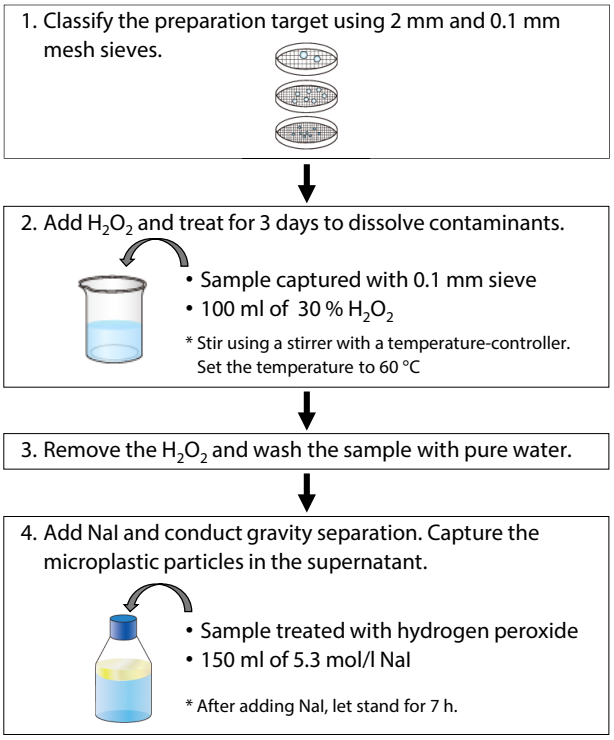

Fig. 5 Workflow of Sample Preparation

Fig. 6 Example of Captured Microplastics

■ Analysis of Microplastics by FTIR

The captured microplastic particles were air-dried, and an analysis was conducted using the Shimadzu Plastic Analyzer plastic analysis system. Plastic Analyzer is a specialized product for evaluation of degradation of plastics, and includes the IRSpirit Fourier transform infrared spectrophotometer, QATR™-S single-reflection ATR accessory, and Plastic Analyzer method package (UV-damaged plastics library, thermal-damaged plastics library, and, analysis programs/method files for IR Pilot™). This system is a useful tool for correctly analyzing the presence/absence of degradation in plastic products and identifying the degradation process, and does not depend on the experience of the analyst. It can also be applied to analyses of microplastics. Product details and examples of use may be found in Application News No. A647.

One analysis result of the captured microplastics will be introduced here. Fig. 7 shows a stereoscopic microscope image of the sample appearance, Table1 shows the measurement conditions, and Fig. 8 shows the measurement results by FTIR.

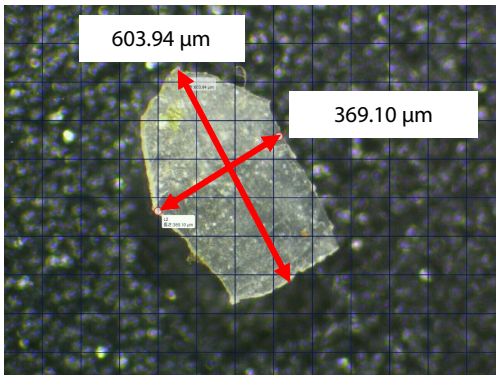

Fig. 7 Stereoscopic Microscope Image of Sample Appearance

Table 1 Measurement Conditions

Instruments	: IRSpirit QATR-S (diamond prism)
Resolution	: 4 cm^{-1}
Accumulation	: 45 times
Wavelength range	: 4000 - 600 cm^{-1}
Apodization function	: SqrTriangle
Detector	: DLATGS

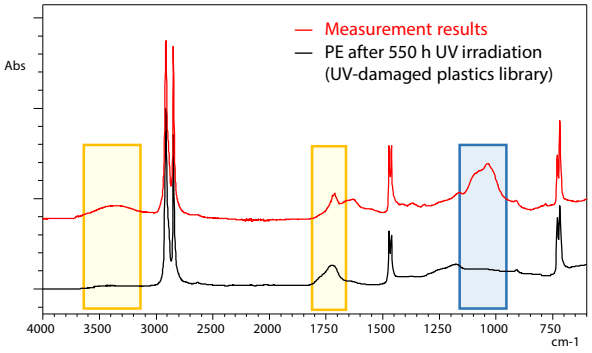

Fig. 8 Measurement Results by FTIR

The UV-damaged plastics library of Plastic Analyzer contains infrared spectra for 14 types of plastics which were subjected to UV irradiation for times of 0 h (no irradiation) to a maximum of 550 h (equivalent to UV exposure for approximately 10 years) using a super accelerated weathering chamber manufactured by Iwasaki Electric Co., Ltd. A hit for polyethylene (PE) after UV irradiation for 550 h was obtained from this library. Because the measurement detected absorption due to O-H stretching vibration at around 3400 cm^{-1} and C=O stretching vibration at around 1750 cm^{-1}, as shown by the yellow boxes in Fig. 8, it can be estimated that this microplastic was affected by oxidative degradation due to exposure to UV radiation in the environment. In addition, the peak at around 1050 cm^{-1} (indicated by the blue box) suggests the possibility of silicate contamination.

■ Conclusion

It was possible to determine that microplastics sampled from rivers had been degraded by exposure to UV radiation by a simple procedure using the Plastic Analyzer plastic analysis system.

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SHIMADZU
Shimadzu Corporation
Analytical & Measuring Instruments Division
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