

Investigation of Reducing Measurement and Analysis Time in Microscopic FT-IR Analysis of Microplastics in Low-Suspended Solids Water, Compliant with ISO 16094-2.

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1. Introduction

Microplastics (MPs) have been detected in drinking water and environment water, raising concerns regarding environmental contamination and potential human exposure. As regulation and monitoring activities expand worldwide, there is an increasing demand for reliable and standardized analytical methods. ISO 16094-2 provides methods for MPs analysis using μ FTIR and μ Raman spectroscopy. However, measurement and data analysis take time-consuming. This study evaluated approaches to improve analytical accuracy and reduce analysis time in ISO 16094-2-compliant μ FTIR analysis.

2. Method

1 L of tap water was subjected to vacuum filtration to collect the sample on a stainless-steel mesh filter. Hydrogen peroxide was added dropwise to the filter, and the assembly was left to stand overnight. A PF (Particle Filter) Holder (Fig. 1) was used to secure the filter. Particle analysis was performed using μ FTIR AIMSight™ and IRXross™ (Fig. 2) and data analysis was conducted using AMSolution™ (Shimadzu Corporation).



Fig. 1 Picture of PF Holder



Fig. 2 Picture of AIMSight and IRXross

3. Evaluation of PF Holder

The PF Holder improved filter flatness and expanded the focus area in microscope images. Representative data and images are shown in Fig. 3 and 4. The spectrum match score improved from 699 to 777. It was demonstrating enhanced particle visibility and identification accuracy using PF Holder.

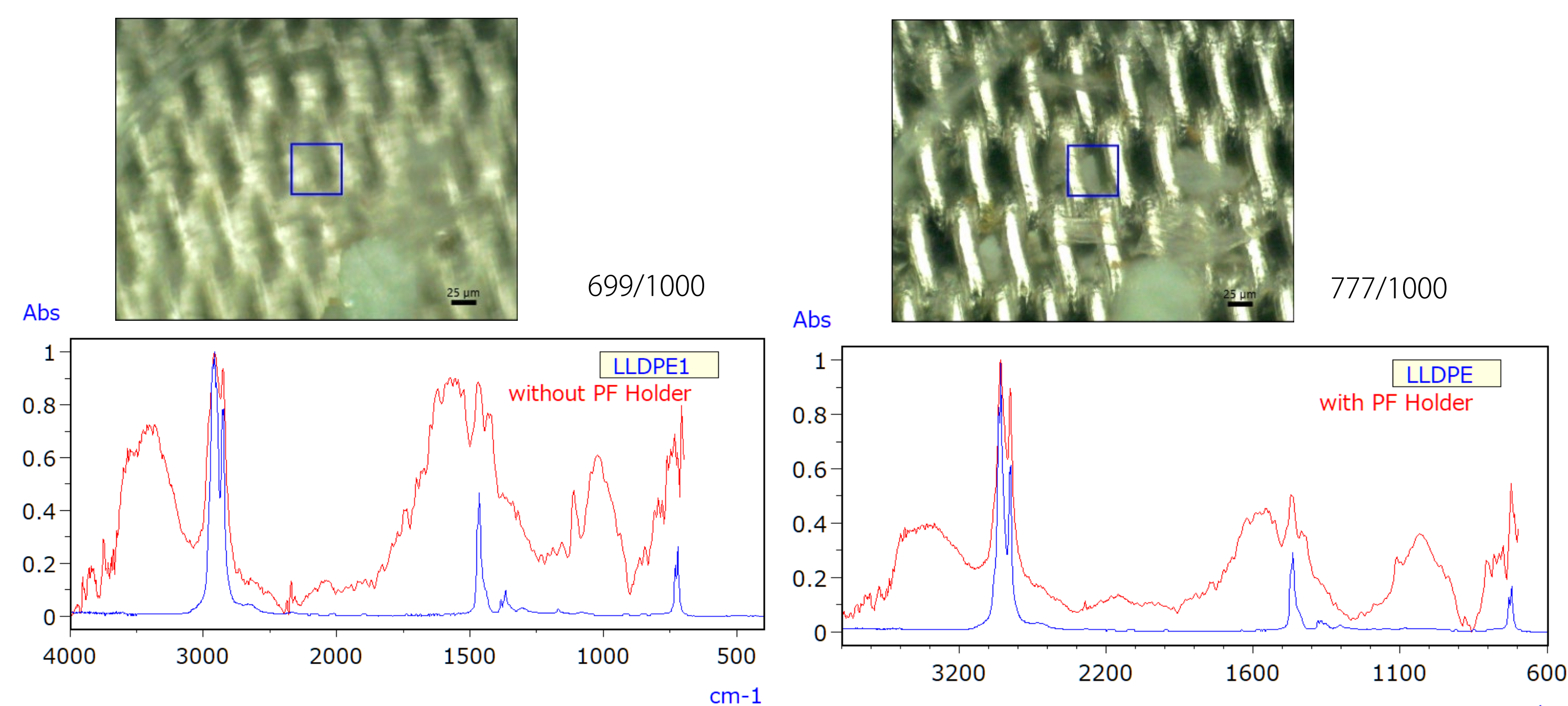


Fig. 3 Infrared Spectrum and Search Results without PF Holder.

Fig. 4 Infrared Spectrum and Search Results with PF Holder.

4. Evaluation of Measurement Time in Mapping Measurement

High-speed Mapping measurement is performed using the specified number of scans, only at the points where the presence of MPs is confirmed from the spectrum. On the other hand, this method skips the measurement points judged to have no sample (Figure 5). For the approximately 4 mm square measurement area shown in Figure 6, the measurement time was reduced from 13.5 hours to 3.5 hours, achieving a 74% reduction. Representative microscope images and extracted infrared spectra are shown in Fig. 7.

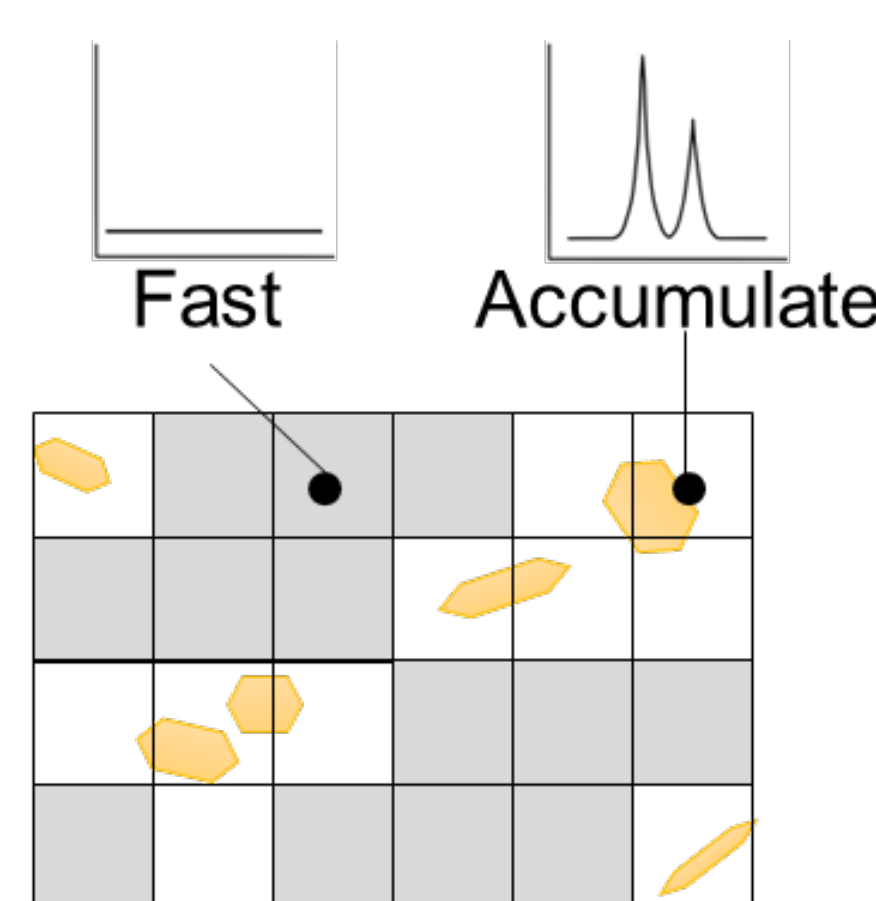


Fig. 5 Schematic Diagram of a High-Speed Mapping Program

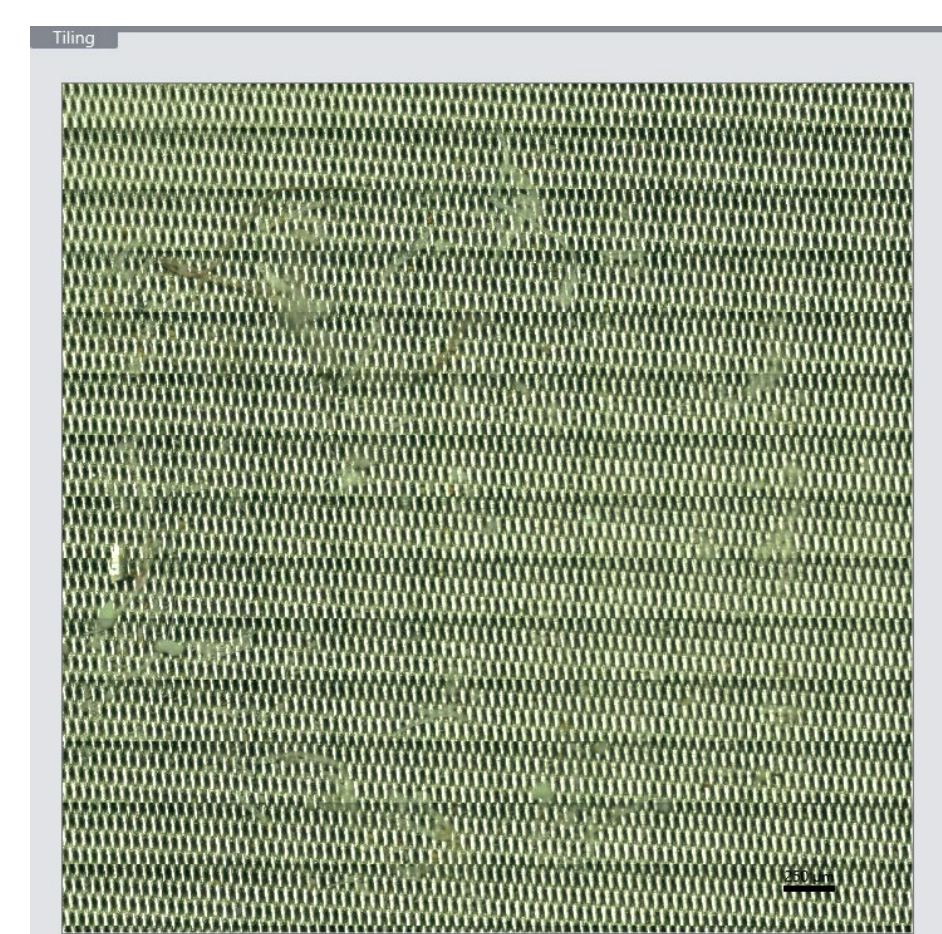


Fig. 6 Measured Microscopic Image

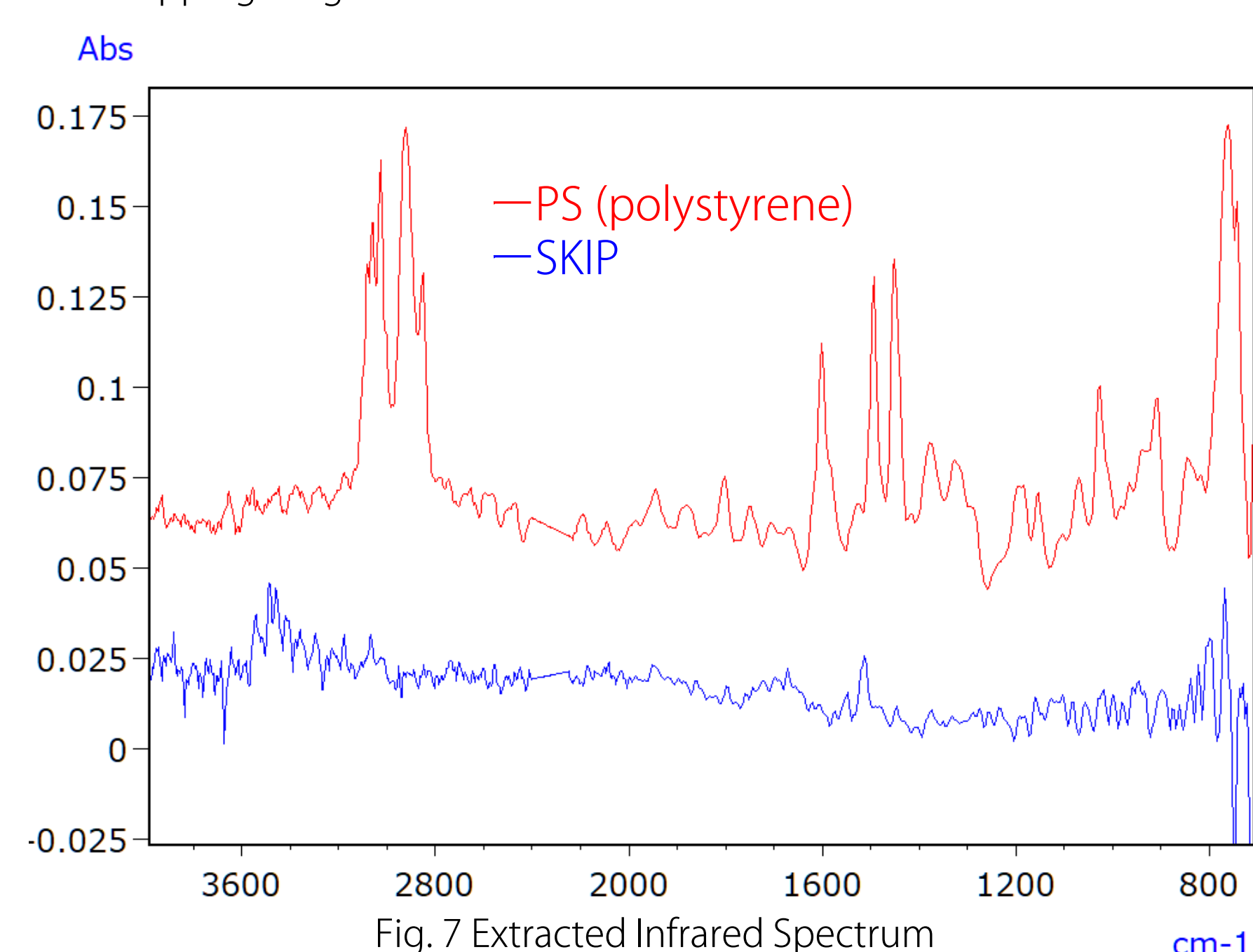


Fig. 7 Extracted Infrared Spectrum

5. Streamlining Particle Analysis for MPs Particles and Results

Using a particle analysis program, the polymer was identified, and the major axis and mass²⁾ of the particles were analyzed simultaneously. Fig. 8-10 shows representative infrared spectra, compositional image, and particle analysis results. A total of 136 particles were detected. While analyzing each particle would typically take 1 min—resulting in a total of 136 min—the analysis was completed in just 3.5 min this case, achieving a 97% reduction in analysis time. Furthermore, number density and mass density were calculated based on the results of the particle analysis; the values were found to be 136 particles/m³ and 0.01 mg/m³, respectively. Qualitative analysis of the plastic was performed using the standard equipped analysis methods, and PP, PE, Nylon 6, PS, PET, and EVA were detected. In terms of particle count, Nylon 6 was detected most frequently, followed by PS and PE.

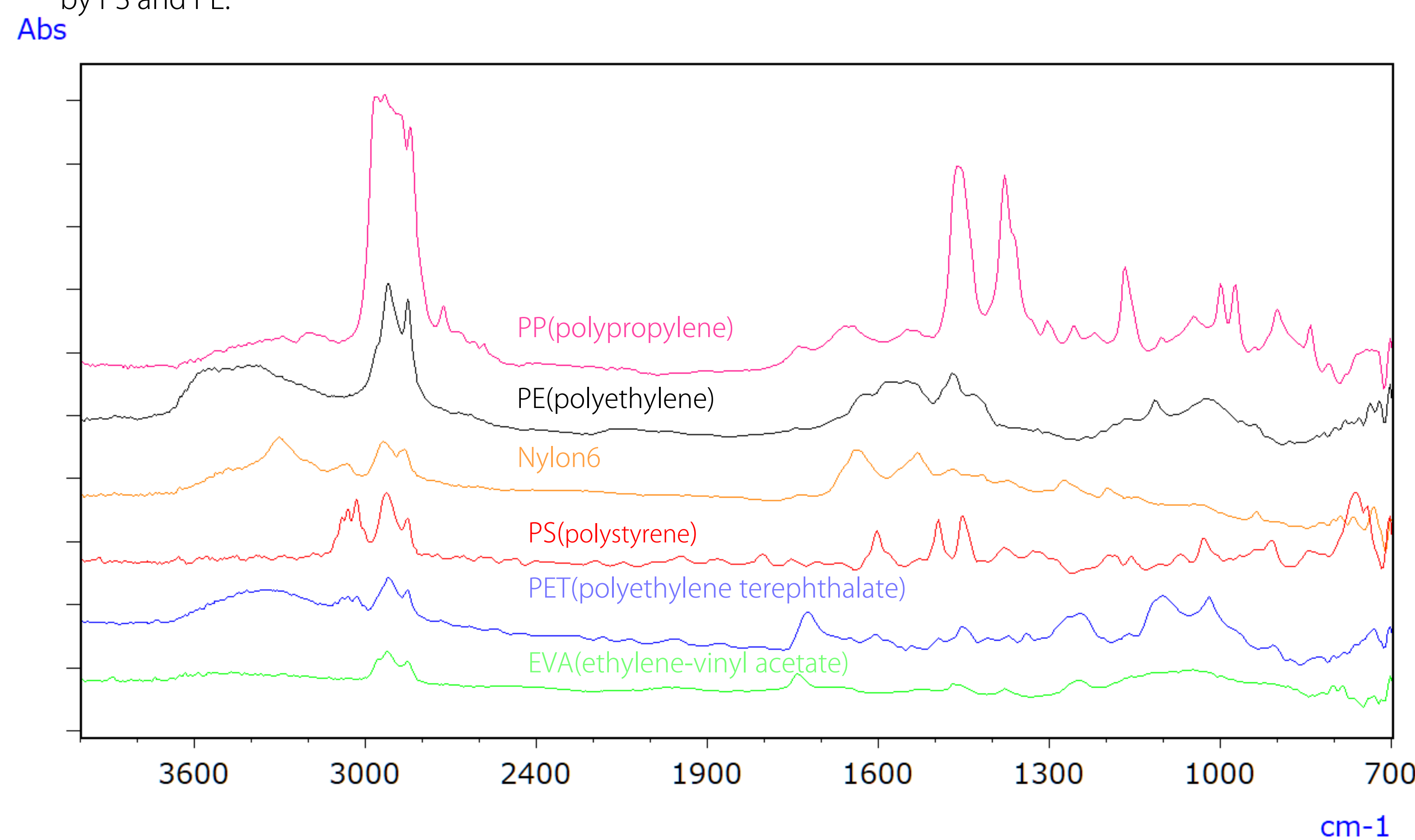


Fig. 8 Infrared Spectrum of each Component

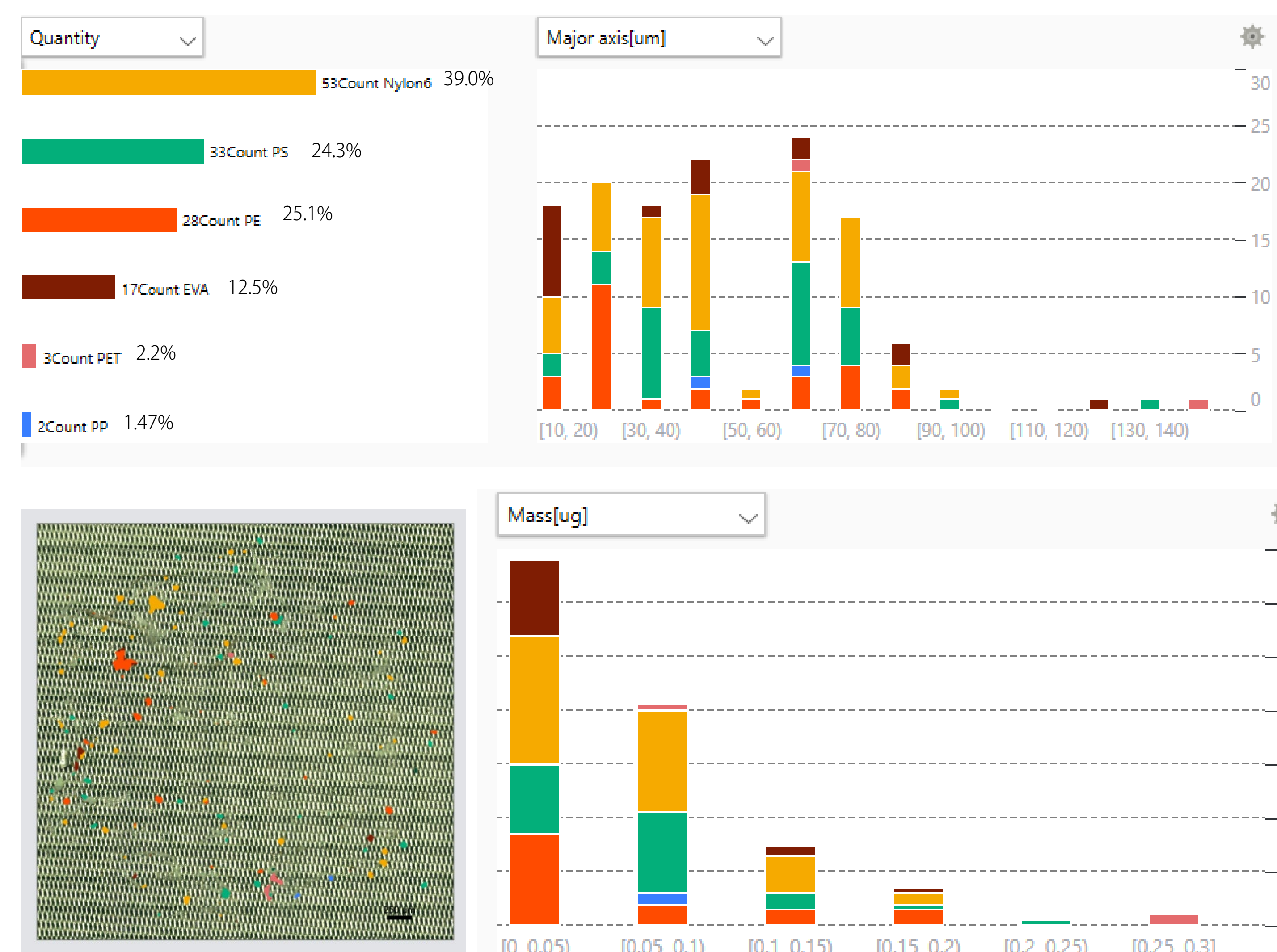


Fig. 9 Microscopic Images Color-Coded by Component

Fig. 10 Part of the Particle Analysis Results

In areas where the sample is present but no color is displayed, cellulose or protein has been detected. Although the pretreatment appears to have been insufficient, this program was still able to effectively detect only the plastic.

6. Conclusion

The PF holder improved measurement accuracy, while high-speed mapping reduced analysis time. Automated particle analysis enabled rapid identification, sizing, and mass evaluation of microplastics. In particular, the system facilitates acquisition of mass data, which is essential for toxicity assessment, and is expected to contribute to future microplastics research.

Reference

- ISO 16094-2:2025 Water quality — Analysis of microplastic in water
Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water
- Kataoka et al. (2024) Geometric relationship between the projected surface area and mass of a plastic particle. Water Research, vol. 261, 122061



Access here for detailed information about Shimadzu's application of MPs.