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Microplastics Monitoring in Japanese River Samples Using an Automatic Sample Preparation Device

Yoshio Ikezawa[1], *Ruth Marfil-Vega* [2],

[1] *Shimadzu Corporation, Kyoto, Japan,* [2] *Shimadzu Scientific Instruments Columbia, MD, U.S.A*

1. Introduction

The occurrence of microplastics (MPs) and nanoplastics (NPs) in the environment is global issue that requires the collaboration of multiple stakeholders to ensure the life cycle of plastics is properly monitored and managed. Most international efforts have focused so far on the standardization and validation of detection techniques, including Raman and FTIR spectroscopy and Py-GCMS. However, now that regulations for MPs are being developed and more environmental laboratories are starting their routine analysis, effective sample preparation is crucial for their success. Current approaches for eliminating organic content from samples or for separating inorganic materials rely on manual digestion and density separation, and the performance of these steps depends on the operator's proficiency. Fig. 1 shows the flow for selecting the instruments according to the analysis purpose. This presentation provides an overview of a universal sample preparation process using the automatic preparation device, MAP-100, and shows the suitability of this device for MPs analysis with the monitoring results of Japanese river samples.

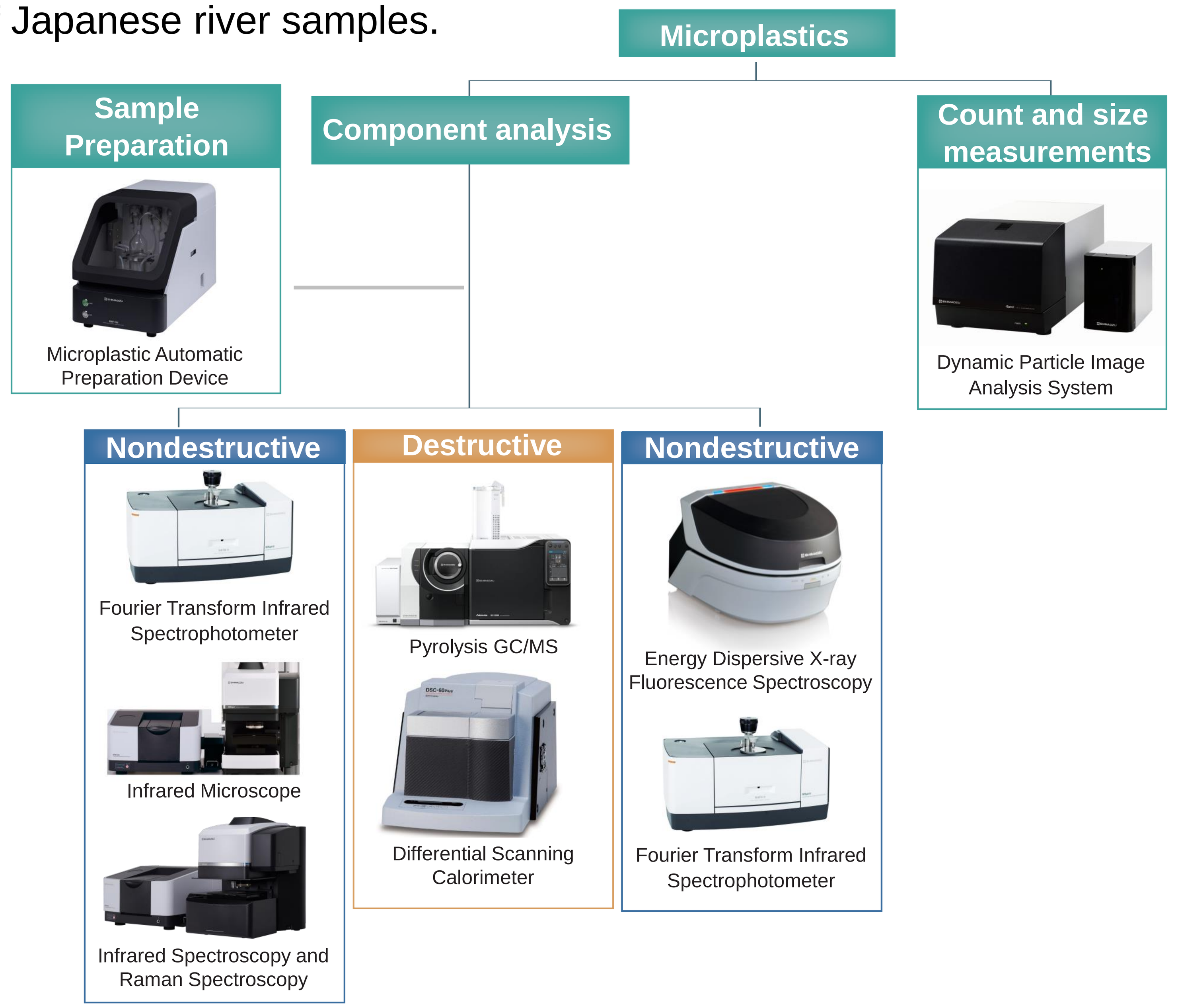


Figure 1 The flow for selecting the instruments according to the analysis purpose

2. Automatic Sample Preparation Device

Japan Environmental Technology Verification

ETV Ministry of the Environment

http://www.env.go.jp/policy/etv/

Figure 2. MAP-100 (certified technology under ETV program from Japan Ministry of Environment)

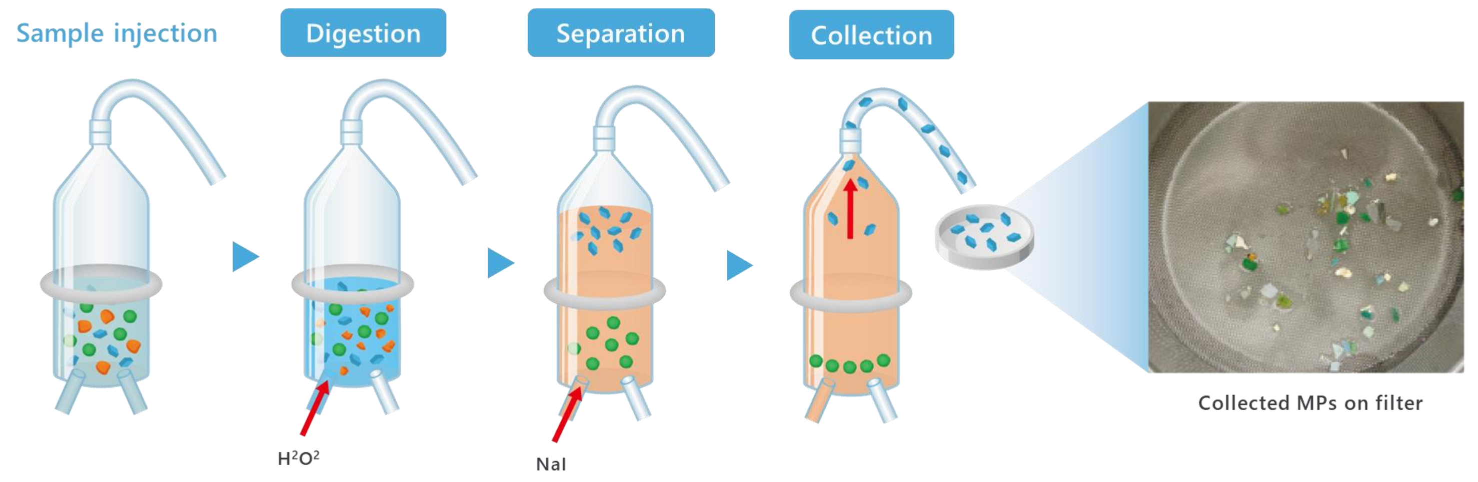


Figure 3. Sample preparation process using MAP-100

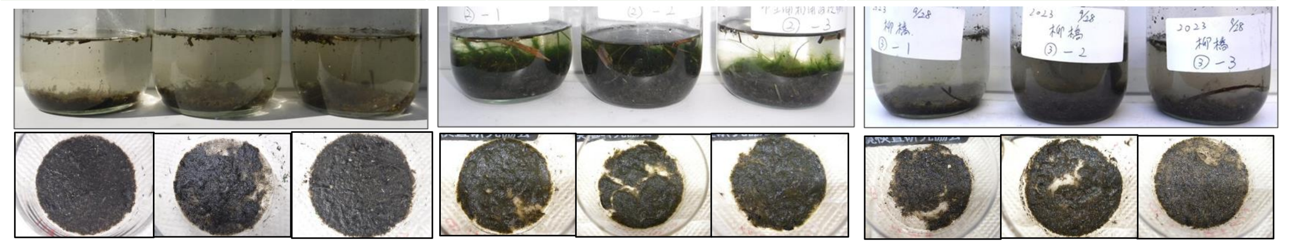
The surface water sample is placed inside the reaction vessel in the automatic preparation device. The control software is used to configure the conditions for each preparation step: digestion, separation, and overflow. Sample automatically undergoes the 3 preparation steps, as shown (Figure 3.). Current configuration of the system allows for the collection of MPs >300 µm, in accordance with main guidelines for sampling techniques of surface water in Japan, using manta net, neuston net or plankton net.

In the Environmental Technology Verification program (ETV program), a reliable third-party organization (Verification organization) who is neither an environmental technology developer nor a user verifies the environmental conservation effects of environmental technology. Furthermore, the results of Verification tests will be widely announced on the Ministry of the Environment website, etc. to support the spread of environmental technology. The verification report of MAP-100 was also certified this year.

3. Experimental Plan

Table 1. River sample information

River	River A			River B			River C		
Point	Location①			Location②			Location③		
date	Sep. 28th 2023			Sep. 28th 2023			Sep. 28th 2023		
Temp.(℃)	30.5			27.0			30.0		
Sample	①-1	①-2	①-3	②-1	②-2	②-3	③-1	③-2	③-3
Volume(m^3)	8.4	9.3	6.8	7.3	10.5	8.5	13.0	18.0	15.0



Nine (9) samples were collected using the plankton net (pore 300 µm) from 3 different rivers (A, B and C) in Japan, according to the guideline published by the Ministry of the Environment, Japan. Performance was evaluated using 2 types of reference particles (PE, 500-600µm) with different densities (1.0 and 1.35 g/cm^3) were evaluated. Twenty-five (25) pieces of each reference particles were added to the river samples before starting automatic sample preparation.

Native and spiked MPs were analyzed after automatic sample preparation. Spiked reference particles were counted on the collection filter. Additional MPs were analyzed by FTIR with Shimadzu original spectroscopic library.

4. Results

Table 2. Results of spike & recovery test of reference particles.

Sample	Weight (g)	Particle Density	Added Number	Recovered Number	Lost Number	Recovery (%)
①-1	4.38	1.00	25	23	2	**92**
		1.35	25	25	0	**100**
①-2	3.67	1.00	25	25	0	**100**
		1.35	25	25	0	**100**
①-3	6.72	1.00	25	23	2	**92**
		1.35	25	25	0	**100**
②-1	10.2	1.00	25	25	0	**100**
		1.35	25	25	0	**100**
②-2	9.88	1.00	25	23	2	**92**
		1.35	25	24	1	**96**
②-3	8.51	1.00	25	23	2	**92**
		1.35	25	24	1	**96**
③-1	5.27	1.00	25	24	1	**96**
		1.35	25	25	0	**100**
③-2	9.36	1.00	25	25	0	**100**
		1.35	25	24	1	**96**
③-3	6.7	1.00	25	23	2	**92**
		1.35	25	25	0	**100**

Table 2 shows the results of spike & recovery test, which are higher than 80% in all sample. This confirms the excellent performance of the MAP-100 even for highly contaminated samples. Table 3 shows the number of collected native MPs in each river samples and Figure 4 shows some photos taken by microscope. MPs present were made of 6. Figure 5 shows the composition based on polymer type of all MPs present in the 9 river samples (without including the spiked reference particles). PE and PP were the major components, (70%). Other polymers identified in most samples include PS and EVA.

Table 3. Number of natural microplastics collected from river sample.

Sample	Natural microplastics (piece)
①-1	11
①-2	27
①-3	27
②-1	34
②-2	55
②-3	53
③-1	57
③-2	119
③-3	90

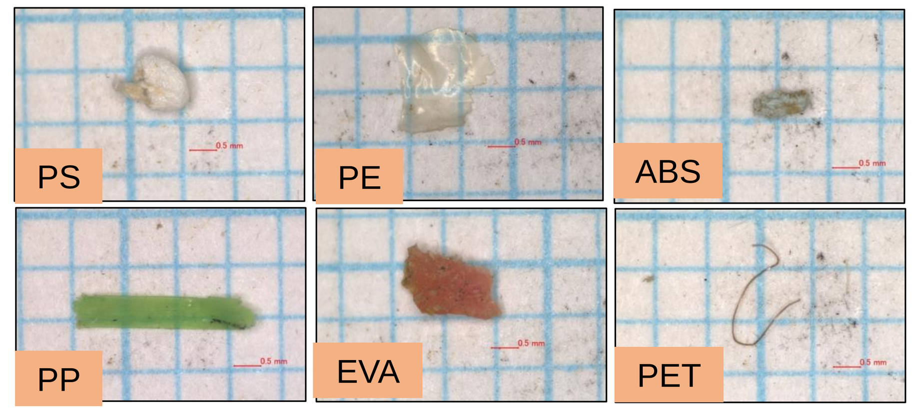


Figure 4. Microscope photos of native microplastics

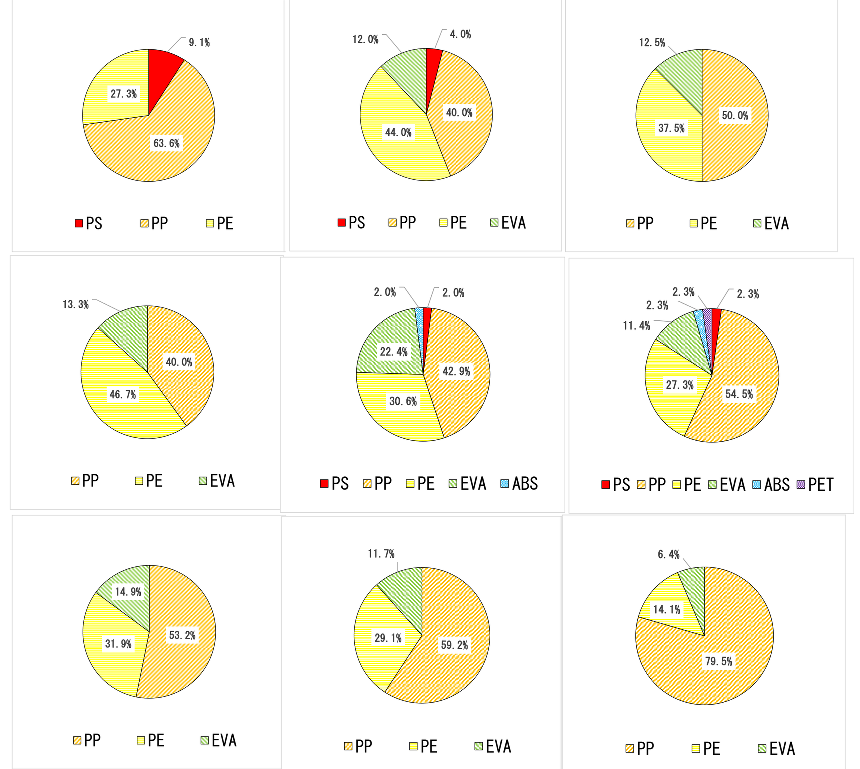


Figure 5. Composition based on polymer type in the 9 river samples

5. Conclusions

Japan Ministry of Environment operates and manages an Environmental Technology Verification (ETV) program, similar to the ETV program from EPA. A third-party organization verifies that the proposed technologies present environmental improvement effects or environmental conservation effects, and advanced measurement technologies related to the environment. The results presented in the poster are those used to obtain the successful verification for the MP-100 in 2024.

References

1. *Guidelines for River Microplastic Monitoring Methods Marge July2021, https://www.env.go.jp/en/water/marine_litter/guidelines/Guidelines_for_River_Microplastic_Monitoring_Methods_Marge.pdf*