

Technical Report

Using an Automatic Preparation System to Collect 100 μm Microplastics from Environmental Surface Waters

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Abstract:

Various steps are required in order to investigate the presence of microplastics in the environment, such as sample acquisition, sample preparation, data acquisition, and data analysis. In particular, pretreating acquired environmental samples is very important for subsequent analysis steps, but currently study results are highly dependent on who performs the preparation steps. Therefore, the MAP-100 microplastic automatic preparation device was designed to standardize preparation processes and improve reproducibility. Previous models could only process 300 μm or larger particles, but due to the growing interest in smaller particle sizes, Shimadzu developed preparation technology that enables the MAP-100 to collect particles down to 100 μm.

Keywords: Microplastics, Automatic preparation, Microparticles, Plastic particles, Environmental pollution

1. Background

Environmental pollution by microplastics is being investigated and study results are being reported in regions around the world. Studies began with investigating drinking water and water resources, but are now expanding to air and soil applications. The key technologies necessary for qualitative and quantitative analysis of microplastics are increasingly becoming internationally standardized. Sample preparation is essential for analyzing microplastics in environmental samples, but unification of preparation methods has been slower to occur than for analytical technologies. Therefore, Shimadzu developed the MAP-100 microplastic automatic preparation device in order to standardize preparation methods and improve reproducibility.

The MAP-100 product specifications were designed to automate the techniques specified in the survey guideline issued by the Japanese Ministry of the Environment, developed with participation by foreign researchers. That survey guideline specifies using a sampling net with a 333 μm pore diameter for sample acquisition and targets particles with a 300 μm or larger diameter for analysis. Consequently, the standard specifications for the MAP-100 are intended for collecting 300 μm or larger microplastic particles. However, in recent years, the scope of research has been expanding from the environmental effects of microplastics to their effects on humans and studies have been focusing on increasingly microscopic particles. More and more cases of environmental survey research have been focusing on finer microplastics. Therefore, to increase the practical applicability of automatic preparation technologies, Shimadzu developed technology that enables using the MAP-100 system to collect 100 μm size microplastics.

2. Instrument Overview and Applicability for 100 μm Microplastics

Microplastic automatic preparation devices are mainly configured from a reagent supply unit, reaction processing unit, collection unit, and a liquid waste processing unit. (Figure 1)



Figure 1: Main Units in MAP-100 Systems

Of the units indicated above, the reaction processing and collection units are the most closely related to the size of microplastics targeted. The new system configuration that enables collection of 100 μm particles was achieved by changing the stainless steel strainer used in the reaction processing unit of the previous model and by changing how the collection unit works. The MAP-100 process flow consists of a decomposition process, separation process, overflow process, and particle collection process. To achieve the 100 μm specification, the separation solution from the overflow process is discharged directly into glass containers. Then the collected solution must be filtered to extract only the particles being analyzed. Examples of using the MAP-100 system with 100 μm capability to process environmental water samples are described below.

3. Processing Seawater Samples—Example 1

3-1. Sample Preparation

1000 L of seawater was pumped from a shallow area of the seashore in Okinawa and filtered through a plankton net. The matter accumulated at the bottom of the tube was used as the sample. The solid sample was placed in the stainless steel strainer inside the reaction vessel of the MAP-100 system (with 100 μm capability). (Figure 2)



Figure 2: Photographs of the Seawater Sample and the Sample in the Strainer

3-2. Preparation

For automatic preparation using the MAP-100, parameters can be specified for the decomposition process, separation process, and overflow process. The preparation parameter settings used for the test are indicated in Table 1. Since the samples were acquired from the sea after a typhoon had passed, the samples contained many organic substances, but the results show how the substances were slowly decomposed by the hydrogen peroxide (Figure 3). After the decomposition process, aqueous sodium iodide solution was injected via the bottom of the reaction vessel to separate microplastics and other light residual contaminants based on specific gravity. Lastly, an overflow process was used to discharge the supernatant from the reaction vessel directly into a glass container. (Figure 4)

Table 1: Automatic Preparation Parameters

Decomposition Process (30 % Hydrogen Peroxide)	Separation by Specific Gravity (Aqueous Sodium Iodide Solution with 1.6 Specific Gravity)
Processing Time : 3 days	Standing Time : 3 hours
Stirring Speed : 200 rpm	Stirring Speed : 500 rpm
Digestion Temp. : 60 °C	Overflow Count : 3 times



Figure 3: Decomposing the Sample (About 3 Days)

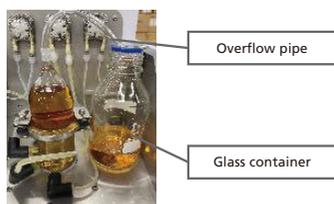


Figure 4: Collecting the Aqueous Sodium Iodide Solution

Because the microplastics are contained in the aqueous sodium iodide solution discharged from the collection container, additional manual steps are required to extract only the microplastics. In order to analyze the microplastics with an infrared microscope (IRXross + AIMsight™), the particles were filtered through a 90 µm mesh stainless steel sieve. Then the solid particles collected on the sieve were recovered with purified water and filtered through a 25 mm OD PTFE filter (with 5 µm pore size) for use as the sample for qualitative analysis. The sieve filtration process is also useful for removing unnecessary residual microparticles in order to prevent the filter from becoming clogged.



Figure 5: Filtered Sample from Collected Solution

4. Processing Seawater Samples—Example 2

This example describes processing a different type of sample than Example 1. At a fishing port in Okinawa, 1000 L of seawater was passed through a plankton net and the substance collected at the bottom of the tube was taken as the sample for automatic preparation using the MAP-100 system with 100 µm capability. Figure 6 shows the sampling site and process. The MAP-100 preparation parameters are indicated in Table 2.



Figure 6: Sampling Site and Process

Table 2: Automatic Preparation Parameters

Decomposition Process (30 % Hydrogen Peroxide)	Separation by Specific Gravity (Aqueous Sodium Iodide Solution with 1.6 Specific Gravity)
Processing Time : 20 hours	Standing Time : 1 hour
Stirring Speed : 200 rpm	Stirring Speed : 500 rpm
Digestion Temp. : 60 °C	Overflow Count : 3 times

In this case, the acquired sample contained less organic matter than the test samples described in the previous section (Example 1), so a shorter decomposition time was sufficient. The decomposition time should be adjusted appropriately depending on the quantity of organic matter contained in the sample. It should be noted that if a large quantity of organic matter remains after the decomposition process, it may lead to variability in subsequent analytical steps and increased analysis time. In this example, results from FTIR and Raman microscope analysis of substances collected during preparation confirmed the presence of PP and PE microplastic particles about 240 µm long and about 120 µm wide. (Figure 7)



PP



PE

Figure 7: Detected Microplastics

Previous MAP-100 models could only collect microplastics 300 µm or larger, but the results confirmed that the updated model can collect microplastics smaller than 300 µm (as small as 100 µm).

5. Conclusion

The lower limit size of particles that can be collected by the MAP-100 system was improved from 300 to 100 µm by changing the parts used in the system.

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