Combining sampling of liquid matrices and an automated sample preparation for microplastic analysis using FPA-µFTIR-Imaging

Lucian Iordachescu, Alvise Vianello, Lasse Abraham Rasmussen, Inga Vanessa Kirstein, Jes Vollertsen

Department of Civil Engineering, Division of Water & Environment, Aalborg University, Denmark

Introduction

- Microplastic (MP) pollution is a global issue affecting almost all types of matrices such as marine waters, freshwaters, wastewaters, etc. In order to quantify the amount of microplastics in a sample using µFTIR-Imaging, unwanted organic matter has to be removed. This is done by procedures including wet peroxide oxidation and enzymatic digestion.
- Typically the sample preparation is done in open laboratory vessels, involving the transfer of samples between vessels and filtering between steps. This procedure does though leave the sample open to the surrounding atmosphere, which increases the risk of contaminating it by airborne microplastics.
- Another issue is that the procedure is labor-intensive and prone to human mistakes. This study proposes a methodology to minimize sample contamination by performing sampling and sample preparation in the same closed device, first opening it when the microplastics concentrate is ready for final analysis. It does so in an automated way, hereby reducing the labor needed to conduct the sample preparation.

Design & Manufacturing

Design in a 3d CAD software

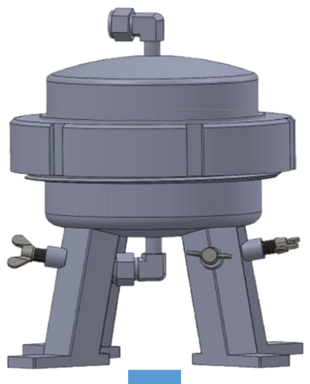

Figure 1 - MP sampling in Greenland

Assembly Animation

Scan the QR code to see a short video of how the device is assembled!

Conceptual model—Sampling

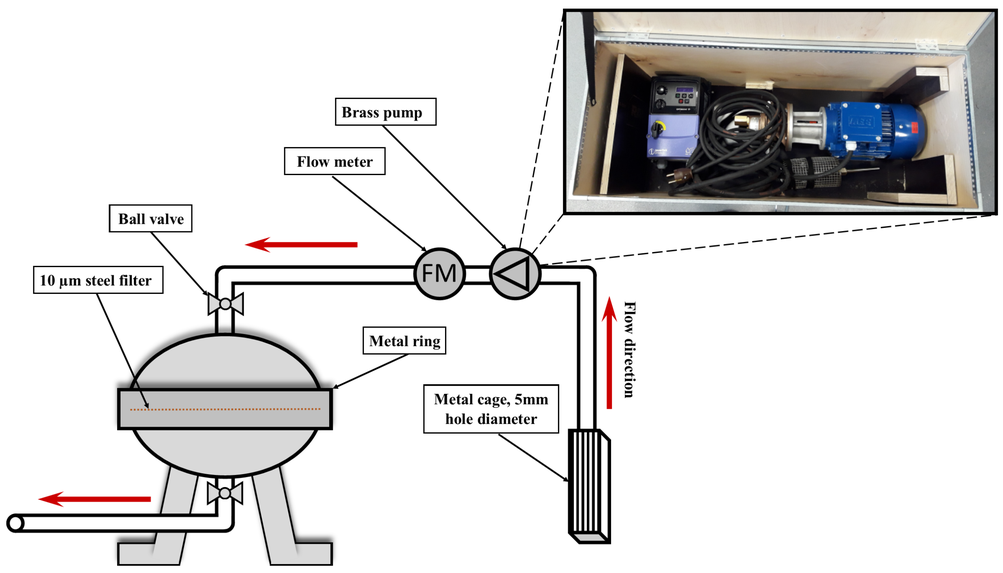

Figure 2 - Schematic of field sampling

Figure 2 illustrates the sampling setup. The liquid matrix is pumped in the device by a brass pump.

The liquid will first pass through a 5mm steel cage and then, the particulate matter is collected on a 10 µm mesh (167 mm in diameter) that is placed inside the device. The amount of liquid that is filtered is monitored by a flowmeter.

After sampling the device can be sealed off by two valves and sent to the laboratory for analysis.

Conceptual model—Sample preparation (Forthcoming)

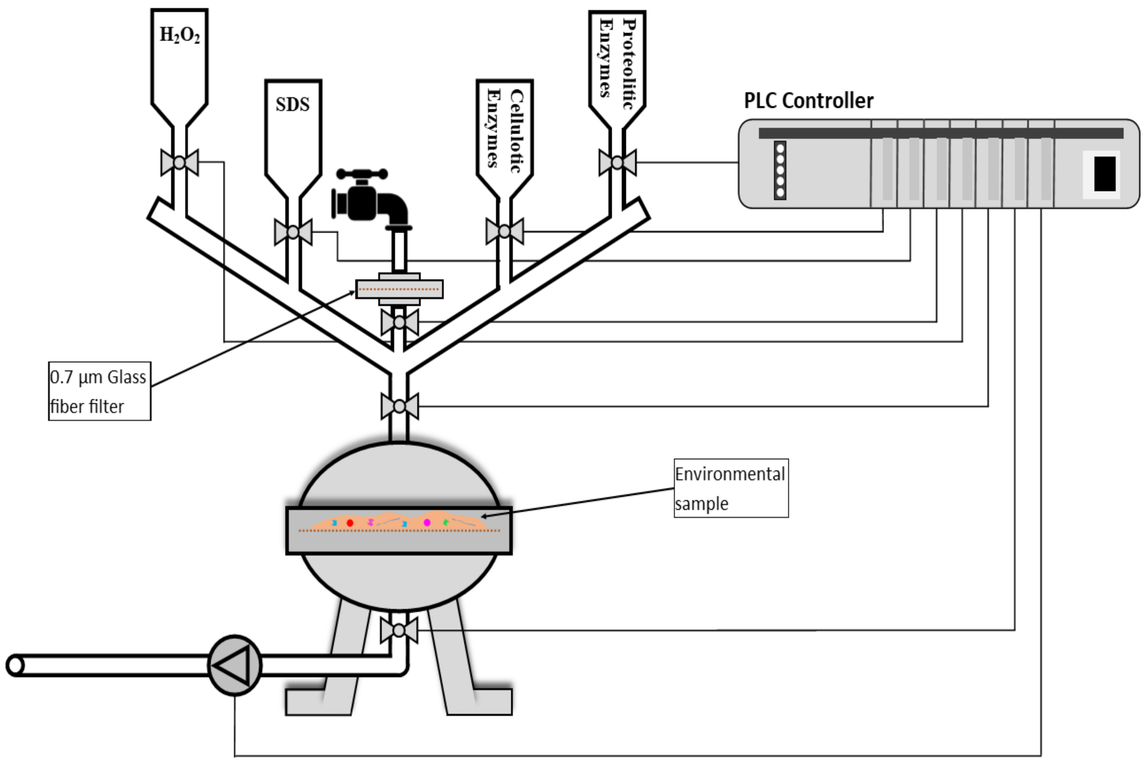

Figure 3 - Schematic of the automated sample preparation

After sampling, the device is placed in a rack and slowly flushed with a sequence of filtered (0.7 µm) treatment solutions: H_2O_2, SDS, cellulotic enzymes, proteolitic enzymes (See figure 2).

Between the steps the filter is flushed with filtered (0.7µm) demineralized water to ensure proper removal of the chemicals. The process is automatized by a PLC-controller in order to minimize the amount of labor put into the sample preparation.

After treatment, the filter is extracted inside a laminar flow cabinet and the particles concentrated. Finally, a sub-sample of the concentrate is analyzed by FPA-µFTIR-Imaging.

Contamination

Contamination rates were tested by analyzing a blank sample (500 mL of Milli-q water) where all the reagents required for the sample preparation (See figure 2) were poured into the device. Afterwards, the filter was taken out from the device and the particles were removed from it using a sonication bath. The particles were then concentrated into 5 mL of EtOH (50% v.v) out of which 1 mL was deposited on a transmissive window and scanned by a Cary 620 FT-IR microscope from Agilent Technologies coupled with a Cary 670 IR spectroscope.

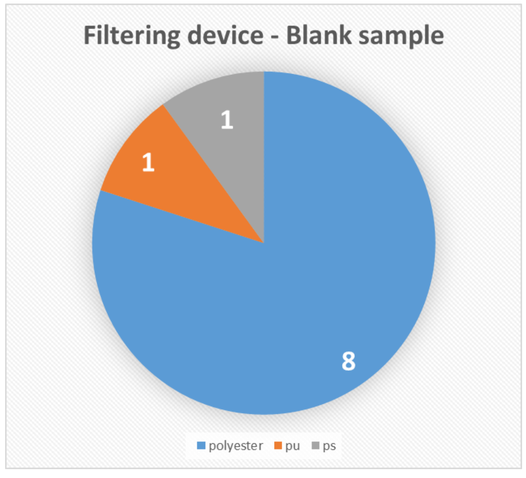

Figure 4 - Pie chart displaying the number and polymer type of the particles identified in the blank sample

Recovery rates

For the recovery tests, 175 particles (100µm diameter PS beads) were pumped in the device, then removed from the filter by sonication. The particles were then concentrated and counted under a stereo microscope.

70 % of the particles were recovered

Conclusion

- The device can filter up to 500L of seawater and up to 300L of stormwater retention pond water per filter.
- Multiple devices can easily be connected together for faster sampling and a bigger volume of water.
- Operating and handling the device is straight-forward and uncomplicated.
- Since the device is entirely made out of stainless steel, it does require some "elbow grease" to handle.

Contact : Lucian Iordachescu - lio@civil.aau.dk
Jes Vollertsen - jv@civil.aau.dk