CHALLENGES OF SAMPLE PREPARATION AND ANALYSIS OF STANDARD SAND AND SEDIMENT SAMPLES IN AN INTERLABORATORY COMPARISM TEST – A PARTICIPANT'S APPROACH

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Introduction & objective

Microplastic (MPs; particles <5mm) have been detected globally in a wide range of environmental elements, but the methods of **sampling, sample preparation and analysis are not harmonised yet**. Different methods apply different steps to clean up the microplastics as much as possible from the matrix of the sample (e.g. sand particles in sediment; biogenic organic matter in soil). For this, usually density separation, oxidation and filtration are applied. There are also a great variety in terms of identification methods. Quantification can either be based on particle number (microscopy/spectroscopy) or total mass (thermoanalytical methods) approach. Due to the variety of methods shown in **Figure 1**, results are not always comparable. In order to avoid this, we **need standardized methods**. Harmonization is facilitated by the implementation of i**nterlaboratory studies (ILS)**, which makes it possible to **compare different laboratories and methods,** such an ILS was organized by the EUROqCHARM.

Materials & methods

- Spiked samples were sent to the participating laboratories. Sand (sample #1) was a blank. Sand (sample #2) was spiked with **PP, PC, PVC** in a size range of **50-299 µm**. Sediment (sample #3) was spiked with **PE, PET, PS** in a size range of **50-299 µm** and **1 mm PE beads.**
- Sediment sample preparation based on **a density separation ($ZnCl_2$) in SVGS device (Figure 2)** and **filtration (Anodisc)** supplemented with an **oxidation (H_2O_2)** step.
- Imaging with ThermoFisher iN10mx **FTIR microscope** was used to detect MPs. The filter was scanned in transmission mode at a pixel resolution of 25 µm.
- Data were analyzed in the siMPle software.

Results & discussion

Based on the submitted results Z-scores were calculated per each polymer type that were added to the samples. A Z-score is a numerical measurement that describes a value's relationship to the mean of a group of values. Z-scores are given to enhance the insights deduced from the ILS and to support the improvements of methodology. We could not detect any MP in the balnk (sample #1) which is a good feedback for us, indicating that **our analytical method does not contaminate the sample.** In case of sample #2, Z-scores were not calculated due to the large standard deviation in the data, which means that we did not receive feedback on the effectiveness of our method in this case. At sample #3 most of **our results were in satisfactory z-scores range** (**Table 1**).

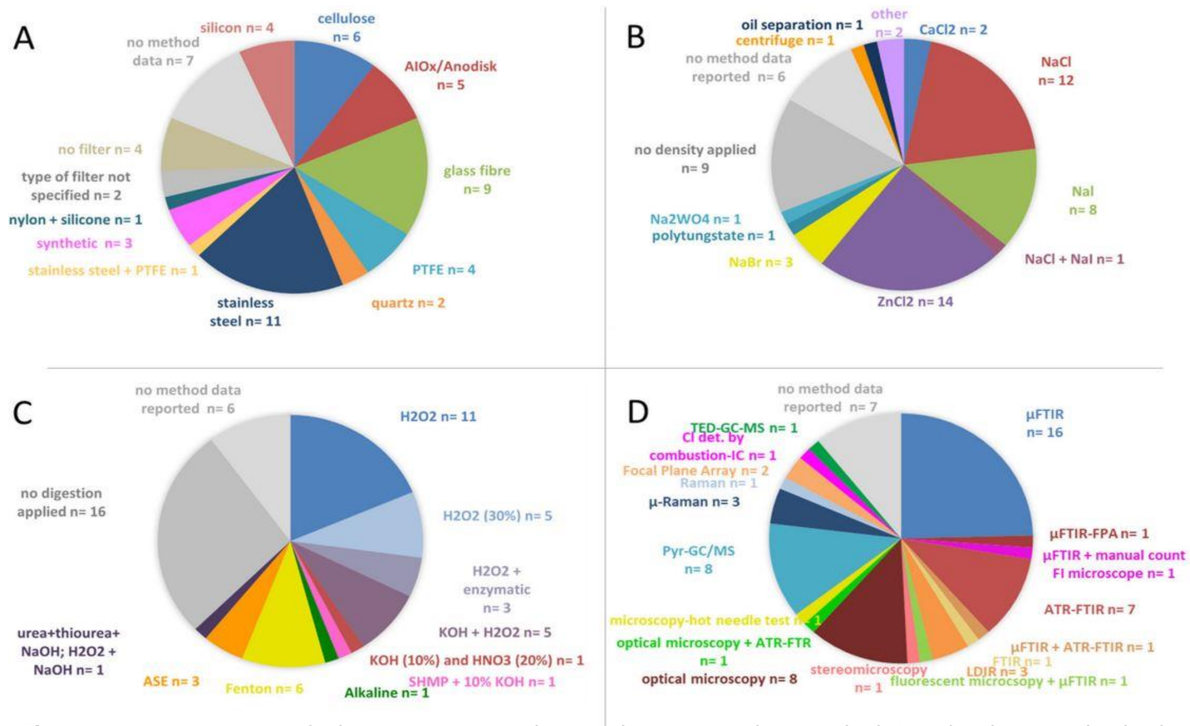

Figure 1.: Summary of data on reported sample preparation and determination methods in EUROqCHARM project for microplastics determination in sediment/sand samples, with A) type of filter used, B) type of salt used, C) type of digestion used and D) type of determination method used.

Labcode	Poly ethylene (300-5000 µm)	Poly propylene (50-299 µm)	Poly carbonate (50-299 µm)	Poly vinylchloride (50-299 µm)
Q4026	-2.65	-1.40	-	-1.87

Table 1: Z-scores calculated for the reported number of the added polymers. Green indicates: Satisfactory performance (|z| < 2); Yellow indicates: Questionable performance (2 < |z| < 3).

An exception to this is the PE in the size range of 300-5000 µm. The reason for this is that this was used in the forms beads, thus was not penetrated by the IR beam used by our technique, as illustrated in **Figure 3.** The use of ATR-FTIR technique can be considered for larger particles**.** This highlights, that **a single method might not cover the entire MPs size range** when larger, thick particles >1mm are considered. This indicates the need not only for the standardised methods, but the refined particle definitions.

Figure 2: Small Volume Glass Separator (SVGS) used for density separation.

Mári, Á., Bordós, G., Gergely, Sz., Büki, M., Háhn, J., Palotai, Z., Besenyő, G., Szabó, É., Salgó, A., Kriszt, B., Szoboszlay, S. (2021): Validation of microplastic sample preparation method for freshwater samples. Water Res. 202. 117409. https://doi.org/10.1016/j.watres.2021.117409

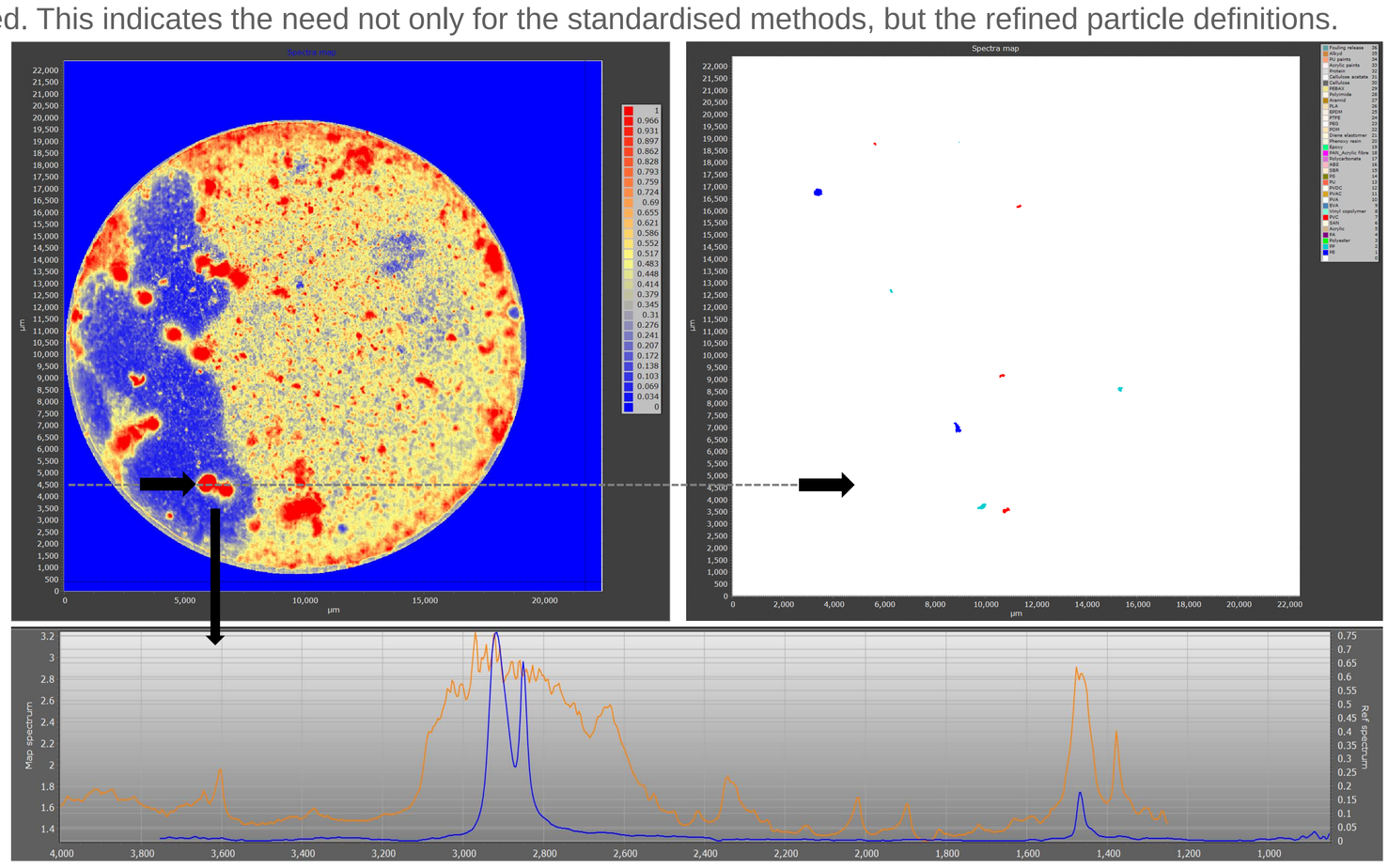

Figure 3: Analysed sediment sample. Heat map (top left total absorbance with red) and the microplastic map (top right, correlation >75%). At the bottom, orange spectrum represents the sample and the blue the PE spectrum from the data base.

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