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MICROPLASTIC RESEARCH AT CEN

Microplastics on the rocks:
adaption of a method for preparation and storage of reference material

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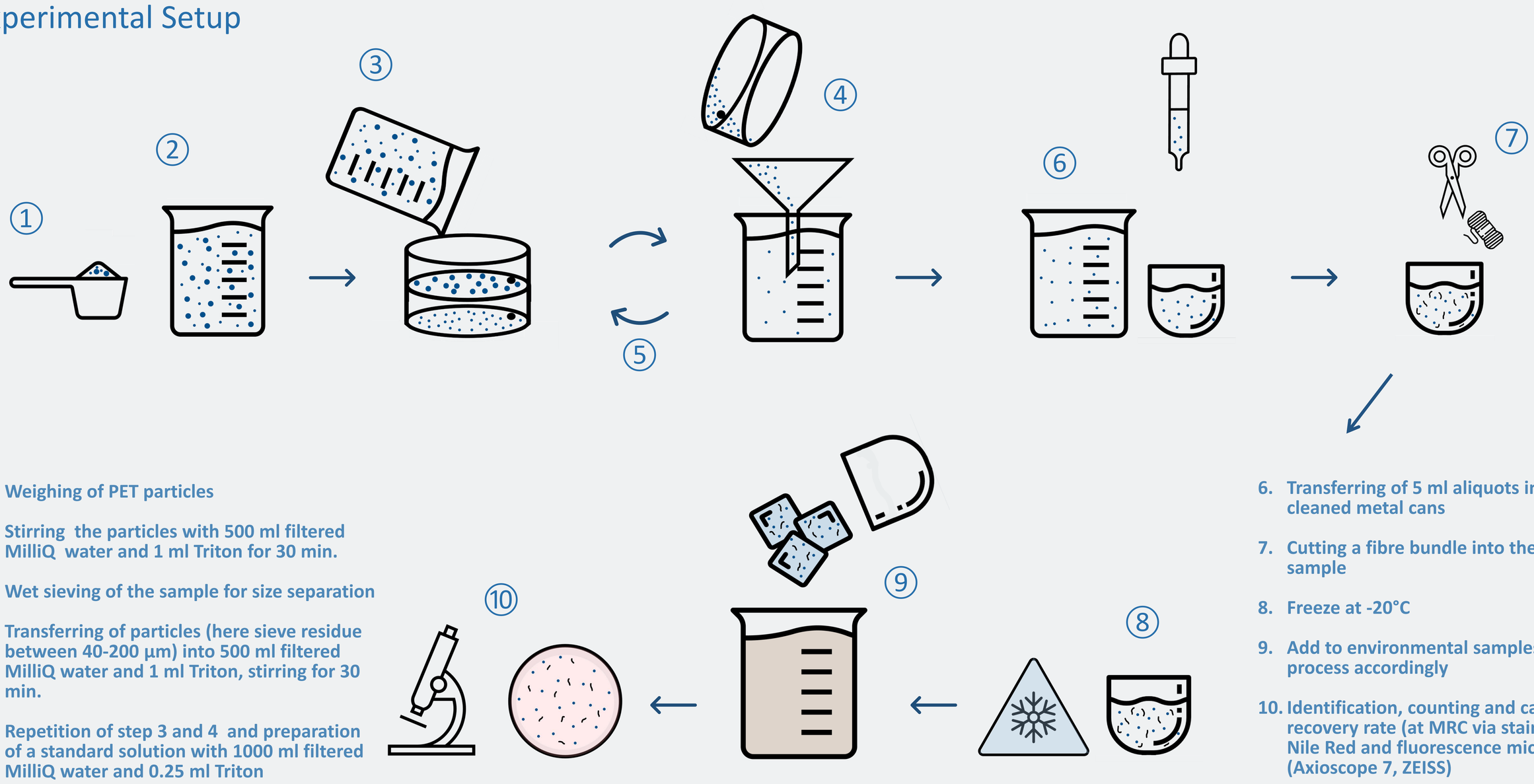
Introduction

Microplastic research and associated sample processing and analytics is a rapidly developing area of research. There are numerous different approaches and methods. These result in the fact that findings are method-specific. Thus results from different studies are comparable only to a limited extent.
The use of reference samples is a prerequisite to check laboratory performances and to assure QA/QC criteria. For this reason, we adapted an approach according to Seghers et al. (2022) to prepare a reference standard solution allowing for adaptions to particle sizes and morphologies.

Material:

- 0.025 g of PET Particles (Goodfellow Cambridge Limited, polyethylene terephthalate max. particle size: 300 µm)
- 2000 ml filtered MilliQ water
- 2.25 ml Triton X-100
- two stainless sieves, : e.g. 200 µm and 40 µm mesh size
- 86 fibres per sample (Goodfellow, Cambridge Limited, Polypropylene, cut fibre bundle)
- 5 ml aliquots transferred into pre-cleaned metal jars

Experimental Setup



1. Weighing of PET particles
2. Stirring the particles with 500 ml filtered MilliQ water and 1 ml Triton for 30 min.
3. Wet sieving of the sample for size separation
4. Transferring of particles (here sieve residue between 40-200 µm) into 500 ml filtered MilliQ water and 1 ml Triton, stirring for 30 min.
5. Repetition of step 3 and 4 and preparation of a standard solution with 1000 ml filtered MilliQ water and 0.25 ml Triton
6. Transferring of 5 ml aliquots into pre-cleaned metal cans
7. Cutting a fibre bundle into the reference sample
8. Freeze at -20°C
9. Add to environmental samples and process accordingly
10. Identification, counting and calculation of recovery rate (at MRC via staining with Nile Red and fluorescence microscopy (Axioscope 7, ZEISS)

Results & Discussion

For the validation of the approach, a total number of eleven samples with PET particles were analysed, five of these samples also contained a fibre bundle consisting of 86 single fibres. In mean, 99.45 ± 8.3 particles per 5 ml aliquots was recorded without an environmental sample matrix such as sediment. As for fibres, an average of 59 ± 8.4 fibres (of 86 added in total) were counted. Based on these values recovery rates are established in order to monitor laboratory processing and performance. The reference samples can either be processed as separate samples parallel to the normal environmental samples or can be added to environmental samples directly before laboratory processing.

Fig. 1: Value distribution of the PET particles and PP fibres

Sample		100 % recovery	99 % recovery	95 % recovery	90 % recovery
PET Particles	Min	91.1	90.11	86.13	81.16
	Mean	99.45	98.46	94.48	89.51
	Max	107.8	106.81	102.83	97.86
PP Fibres	Min	50.51	49.92	47.56	44.61
	Mean	59	58.41	56.05	53.10
	Max	67.48	66.9	64.54	61.59

Tab. 1: Recovery Rates

Table 1 provides recovery rates of the processed reference samples. Mean values were calculated as relative percentage and indicating the standard deviation.

Recommendations and Conclusion

Reference materials should cover the different morphologies, sizes and polymer composition of microplastic particles.
In this experiment we used fibres and fragments, as these represent the largest occurrence in environmental samples. The double sieving represents the targeted size spectrum very well, only 1.4 % of the particles identified were outside this size range. We used mesh sizes between 40 and 200 µm corresponding to the size spectrum of microplastic particles to be addressed in current monitoring projects of microplastics in seabed sediments. However, the size range can be adjusted individually. In terms of polymer composition, this setup covers polymers with high density (PET with a density of 1.38 g/cm^3). In addition, an experiment with LDPE (density 0.93 g/cm^3) is currently being processed.

Summarising, the approach can provide a large amount of reference material that can be prepared according to pre-tests easily and in a timely manner. By freezing in small portions, the reference samples can be used individually and also stored over a longer time period. Sizes, morphologies and material density of polymers can be adjusted independently, as the freezing process has no effect on the reference polymers themselves.
For the time being until commercially referenced material for microplastic analyses is commercially available this provides a good and alternative method for preparing and storing reference material for different environmental matrices.

Literature

Seghers, J., Stefaniak, E.A., La Spina, R., Cella, C., Mehn, D., Gililand, D., Held, A., Jacobsson, U. & H. Emteborg: Preparation of a reference material for microplastics in water—evaluation of homogeneity. Anal Bioanal Chem 414, 385–397 (2022). https://doi.org/10.1007/s00216-021-03198-7

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