A review of techniques for the separation of microplastics from sediment and other media

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Abstract

This paper focused on the extraction efficiencies and methodology of techniques for the extraction of microplastics from sediment. The extraction techniques: density separation and the Munich Plastic Sediment Separator (MPSS) for Large Microplastic Particles (L-MPP) are highly efficient and so is MPSS and elutriation and floatation for Small Microplastic Particles (S-MPP)/ < 1 mm plastic particles. I recommend in this paper that the comparability of data in the same format is essential to knowing which extraction technique works best and when it does.

Density Separation, The Munich Plastic Sediment Separator (MPSS), Elutriation and Density Separation, Chemical Digestion and Froth Floatation.

In (Imholf et al., 2012), the density separation procedure with $ZnCl_2$ (1.6-1.7 kg/L) reported a 99.7 ± 0.6 % recovery of Large Microplastic Particles (L-MPP, 5 -1 mm) and a weight recovery rate of 99.12 ± 3.98% from sediment. An average 39.8 ± 16.6% weight recovery rate for Small Microplastic Particles (S-MPP, < 1 mm) was reported. Results were validated by Raman Microscopy.

Froth floatation of sediment; with one dishwasher tab as surfactant, 625 mg/L pine oil as wetting agent and 0.625 mL/L froth conditioner; reported an overall 55.0 ± 28.8% recovery rate for L-MPP. The recoveries for 8 different plastic types were remarkably different from each other.

Meanwhile, the MPSS with $ZnCl_2$ (1.6-1.7 kg/L) reported a 100 % recovery rate for L-MPP and a weight recovery rate of 95.5 ± 1.8% for S-MPP after three separations each from sediment. The result was validated by Raman Microscopy.

In (Claessens et al., 2013), classical density separation as pioneered by (Thompson et al., 2004) reported extractions of 75% fibres, 61% granules and 0 % PVC particles using NaCl solution (1.2 kg/L) from sediment. The extraction, which was repeated 2 to 3 times, was done for microplastics < 1 mm. Likewise, an elutriation and floatation followed by NaI solution (1.6 kg/L) density separation by centrifugation reported 98% extraction of fibres and 100% extraction of granules and PVC particles. The elutriation step was conducted once and the density separation was completed 2 to 3 times.

Chemical digestion of mussel soft tissue involving 1 h heating at 60°C with cold or warm HNO_3 (22.5M)/ NaOH (52.5M)/ H_2O_2 (32.6M) or 3:1 v/v mixtures of HNO_3: H_2O_2/ HNO_3:HCl (32.3M) followed by boiling at 100 °C for 1 h then dilution (1:10 v/v) with warm (boiling) or cold (room) filtered deionised water followed by filtration over a pre-weighed 5 µm cellulose nitrate membrane filter yielded the highest efficiencies. The most efficient was obtained through heating for 1 h at 60°C with HNO_3 then boiling for 1 h at 100 °C followed by dilution with warm (~ 80 °C) filtered deionised water and filtration. Digestion efficiency was calculated when the filters containing remaining mussel soft tissue had been dried for 24 h at 60 °C and weighed and compared with the pre-chemical digestion weight of the mussel soft tissue. The procedure with the smallest soft tissue fraction remaining on the filter; the most efficient, was validated by spiking 10 and 30 µm polystyrene spheres (PS) into different mussel soft tissues and into a flask. Nylon fibres (100 × 400 µm and 30 × 200 µm) were also spiked into different mussel soft tissues. The PS in the flask after the procedure were melted together. Some PS imbedded in mussel soft tissue were recovered: 65.8% of 10 µm PS and 77% of 30 µm. No nylon fibres were recovered after the procedure. The procedure was updated to overnight destruction with HNO_3 followed by 2 h boiling (~100 °C) and warm filtration at

approximately 80 °C. This enabled these extractions: 93.6% of 10 µm PS, 97.9% of 30 µm PS, 98.3 % of 100 × 400 µm nylon fibres and 0% of 30 × 200 µm nylon fibres.

Discussion.

Density separation of L-MPP is narrowly more effective with the MPSS is suggested by comparing extraction data in (Imholf et al., 2012). The extraction with MPSS is 100% effective after three repetitions. The extraction of density separation is 99.7% after one extraction. It could be possible to extract all microplastics with density separation if the extraction is repeated twice more.

The MPSS had 95.5% weight recovery of S-MPP after three extractions which was a + 55.7% rise in effectiveness from density separation after one extraction with the same fluid and identical sediment. In (Claessens et al., 2013), density separation of S-MPP by centrifugation with a similarly dense fluid after elutriation and floatation reported an average 99.3% extraction efficiency after 2 to 3 extractions. The MPSS and density separation by centrifugation are clearly very good methods but it is impossible for a direct comparison as they both report different recovery methods.

The classical density separation by (Thompson et al., 2004) is bettered for extraction efficiency using elutriation and floatation then a stronger separation fluid in the following density separation by centrifugation.

In froth floatation, L-MPP particles with low density had a high recovery rate while those with high density had low recovery rates. (Imholf et al., 2012) reports that froth floatation is a complex method for plastic particle separation. More studies that have been done will have to be considered to fully evaluate this technique.

2.Methodology.

2.1 Density Separation.

A reaction pipe of 108 cm height and 15 cm diameter with four wooden airstones for aeration at the base below an intermediate floor with a 4 mm pore size was set up in (Imholf et al., 2012). 12 L separation fluid was added. 1 L clean sediment spiked with ten L-MPP for each environmentally relevant plastic type (Imholf et al., 2012) within 2-5 mm size range or 0.1 g of each of the same plastic types for S-MPP was filled into the reaction pipe from the top, by tapping; gently, the glass beaker containing the sample. The sample was aerated for 12 h, then left for 2 h while denser particles settled. The upper 3 L was transferred to a conical flask. The top layer with the plastic particles in the flask was decanted into another flask, reducing the fluid volume and then transferred to a filter holder. The sample was vacuum filtered through a quartz filter paper with a 0.3 µm retention size and rinsed twice with deionised water.

Specifically, for S-MPP, the filter was covered 3 times with 10 mL H_2O_2 (30%) to remove organic material before being washed again twice with pure water and dried at 30 °C.

2.2 Elutriation and floatation

A PVC column of 147 cm length and 15 cm diameter with a 35 µm mesh supported by a 1 mm strong mesh at the base and a 1 mm sieve on top is described in (Claessens et al., 2013). 500 mL sediment spiked with 50 particles or fibres is put in the column through the top sieve, removing < 1 mm debris. The sieve was protected with a cover. An upward water flow (300 L/h) was created by forcing water up the column through an entrance at the base. Aeration was provided to ensure the separation of plastic from sediment with three large air stones. The lighter plastic particles were carried by the water flow to the top of the column, where an opening on the edge with a 38 µm sieve captures them. The water flow was maintained for 15 minutes. The

material captured in the sieve was subjected to 3.3 M NaI extraction by transferring it to a 50 mL centrifuge tube with 40 mL separation fluid. After vigorous manual shaking, 5 min centrifugation at 3500g was done. Post – centrifugation, the top layer containing the microplastics was vacuum filtered over a 5 µm membrane filter. The extraction was done 2 to 3 times.

2.3 MPSS

(Imholf et al., 2012) reports on the Munich Plastic Sediment Separator which is mad of 3 stainless steel components connected with flanges equipped with Viton O-rings. The sediment container, 4-5 cm tall with a 30 cm diameter can analyse a 6 L sample in a single run. A 14 revolutions per min electric rotor for stirring the sediment is at the bottom of the container along with a bottom valve to drain the separating fluid. The standpipe atop the sediment container is conical with a 12 cm diameter at the top to allow for a high concentration of microplastics in an extracted sample volume. The dividing chamber atop the standpipe with a ball valve can be closed by the ball valve and detached from the MPSS with a 68 mL sample volume. An integrated filter holder (47 mm) in the chamber can be used for direct vacuum filtration if the chamber is upside down after the separation. Ventilation of the MPSS sample is controlled by a vent screw.

With the standpipe atop the sediment container, the separation fluid was added to the MPSS to ~ 85% fill height from the top sediment inlet flange. With the electric rotor revolving, the sediment (same as for 2.1 Density separation) was added through the top sediment inlet flange in low feeding rates to allow for initial separation. The density was controlled by the electric rotor. After stirring the sediment for 15 min (alternatively, 12 h is possible) and letting the sediment settle for 1 – 2 h, the dividing chamber was put atop the sediment inlet flange and fresh separation fluid was introduced through the bottom valve so as to elevate the fluid level and lift the floating plastic particles into the dividing chamber through the open ball valve. The ball valve was closed and the fluid level was lowered by opening the bottom valve and the vent screw. The dividing chamber was detached. The process from putting the diving chamber to here was repeated 2 more times. By opening the ball valve, vacuum filtration with a quartz filter (47 mm) was done with rinsing of the filter holder walls three times with pure water. If the vacuum sample had organic impurities, 30% H_2O_2 washing was done before the washings with water. If the vacuum sample was full of organic impurities, 30% H_2O_2 washing facilitated by adding H_2SO_4 was done prior to the washings with water. The filters were dried and stored in PetriSlides™.

Discussion and Conclusion

The important difference within the reported procedures is the type of plastic particles extracted. Both density separation and MPSS extract L-MPP and S-MPP while elutriation and floatation extracts < 1 mm plastic. With the extraction rates reported differently and the S-MPP/ < 1 mm plastic spikes reported differently also, it is impossible to make a comparison. However, two other comparisons are as follows: the density of $ZnCl_2$ (1.6-1.7 kg/L) and NaI solution (1.6 kg/L) are similar but not exact. It is possible that this impacts the extractions a bit. Finally, the cost of each separation fluid; means for financial reasons, the use of $ZnCl_2$ is highly recommended (Thompson et al., 2004).

To conclude, the available techniques of extracting microplastics from sediment are diverse and some are highly efficient by optimisation e.g. floatation and elutriation and MPSS. The available techniques for the extraction of microplastics from biota e.g. mussel, are optimisable as well, however for comparison, a review needs to be done. Although we have good

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techniques for the extraction of microplastics from sediment, if data is not able to be compared, its difficult to see which extraction technique works best when.

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