MICROPLASTICS IN MARINE SEDIMENTS

A COMPARISON OF CURRENT EXTRACTION & ISOLATION METHODS

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ABSTRACT

Despite frequent field observations of microplastics (particles <5mm in size) there is a critical knowledge gap of their fate and effects in marine environments. Many of the microplastics will ultimately accumulate in marine sediments as a result of physicochemical and biological processes. Methods exist for the extraction and isolation of microplastics from marine sediments, but major procedural differences prevent meaningful comparison among methods.

Our goal is to conduct a systematic assessment of five commonly used methods for microplastic isolation and identification in representative marine sediments. We reviewed each method for its applicability in two sediment types (sandy and silty) as well as quantified for their ability to recover microplastics amended into sediment samples. The research will allow for quantification of the performance for five methods, as well as provide initial recommendations for routine microplastic monitoring procedures in marine sediments.

BACKGROUND

- Many microplastics ultimately accumulate in marine sediments.
- Several methods exist for the extraction of microplastics from marine sediments, but major procedural differences have prevented meaningful comparison among methods.
- For this assessment, five methods were reviewed for their applicability using two sediment types as well as quantified on their ability to recover microplastics amended into sediment.

METHODS

- Five types of plastic were chosen (Figure 1): polyvinyl chloride (PVC), polyethylene (PE), polystyrene (PS), polyethylene terephthalate (PET), and polypropylene (PP). Plastics represent an array of sizes, densities, usage, and shapes (Table 1).
- 20+ pieces of each plastic were spiked into each sediment sample (sand or silt) and homogenized on a rolling mill for 48 hours.

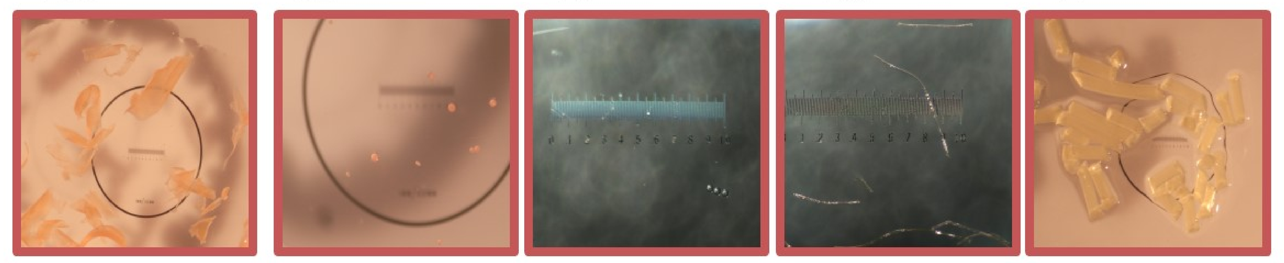

Figure 1. Five plastics used (left to right): polyvinyl chloride (PVC), polyethylene (PE), polystyrene (PS), polyethylene terephthalate (PET), and polypropylene (PP).

Table 1. Properties of plastics used for study.

	PVC	PE	PS	PET	PP
Density g/cm^3	1.35	1.03	0.96	1.38	0.91
Size (µm)	500	125	40	250	700
Shape	flake	sphere	sphere	fiber	fiber
Origin	pipe	pristine	pristine	embroidery floss	rope

- Two representative sediments were used:
- Sandy sediment- d_{90} (90th percentile cumulative particle diameter) = 345.2 µm
  - Silty sediment- d_{90}=62.57 µm
- Both sediments were press sieved through a 2mm sieve.
- Sandy sediment autoclaved at 550°C for six hours to remove organic carbon.
- Five methods were reviewed for microplastic extraction (Table 2).
- 13 samples were evaluated for each method:
  - 5 spiked samples in sandy sediment
  - 5 spiked samples in silty sediment
  - 1 sandy sediment blank
  - 1 silty sediment blank
  - 1 water blank

Table 2. Methods assessed for the study.

Rochman et al.

150g (d) sediment, press sieved >45um → Reconstituted with 1.4g/cm3 $CaCl_2$ → Sorted through 45 and 500 µm sieves → Agitated in beakers with $CaCl_2$ solution → 1-L separatory funnel with $CaCl_2$

Nuelle et al., 2014

1,000g (d) sediment → air induced overflow (AIO) in 2-L beaker with 1.2g/cm^3 NaCl → Decant into 1-L separatory funnel with 1.8 g/cm^3 NaI → Remove organic matter with 35% H_2O_2

Zobkov & Esiukova, 2017

400g (w) sediment → Agitated in beakers with 1.6g/cm^3 $ZnCl_2$ solution → Wet peroxide oxidation with 30% H_2O_2 and Fe(II) → Calcite digestion with 4.5% HCl → Decant into funnel with 1.6 g/cm^3 $ZnCl_2$

Coppock et al., 2017

75g (d) sediment → Agitated in with stir bar in sediment microplastic isolation (Figure 2) unit with 1.5g/cm^3 $ZnCl_2$

Fries et al., 2013

175g (w) sediment → Shaken in 2-L separatory funnel 1.2 g/cm^3 NaCl solution

Figure 2. Sediment microplastic isolation unit, designed by Coppock et al., 2017.

- To minimize contamination, 100% cotton lab coats were worn at all times, and experiments were conducted inside laminar flow hood. Use of plastic equipment was restricted unless necessary.
- All samples were ultimately vacuum filtered through 20um PCTE membrane and visually counted with a Nikon SMZ 745T microscope equipped with an AU-600-HDS camera and digital display.
- Each PCTE filter was inspected visually under the microscope to identify microplastic recovery (Figure 3).
- All filters were counted to determine percent recovery from each method.
- Air blanks were recorded for each sample and quantified using a NightSea UV fluorescence adapter.

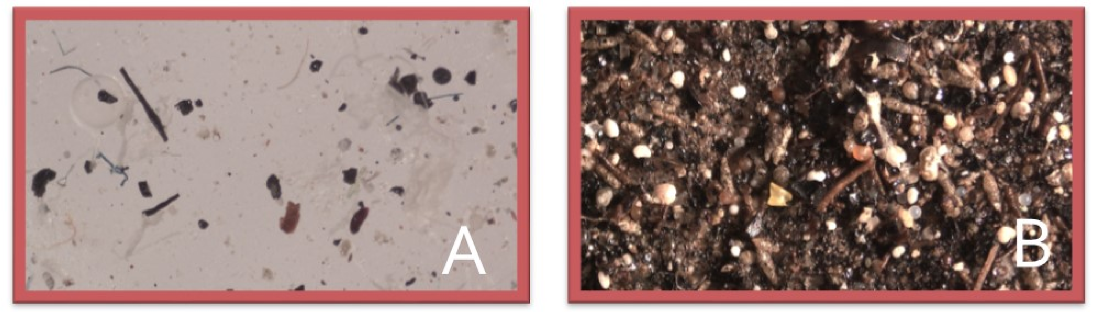

Figure 3. Representative photographs from filters of a sandy (2a) and silty (2b) sediments.

RESULTS

- Methods ranked based on highest recovery per polymer and sediment type (Table 3).
- Samples were compared by plastic types and methods (Figure 4).
- Overall differences (α = 0.05) between plastic types and methods identified by ANOVA were further isolated to specific plastic types and methods using the multiple means comparison procedures.
- Significant interaction differences (α = 0.05) between method and sediment type were evaluated using Bonferroni correction.
- Overall method-specific means were evaluated with Tukey-Kramer analysis (Figure 5).
- Data from Zobkov method is still under review.

Table 3. Quantitative ranking of methods. Method with highest polymer recovery by sediment type.

	PVC	PE	PS	PET	PP
Silt	Rochman	Coppock	Nuelle	Rochman	Coppock
Sand	Coppock	Nuelle	Nuelle	Nuelle	Fries

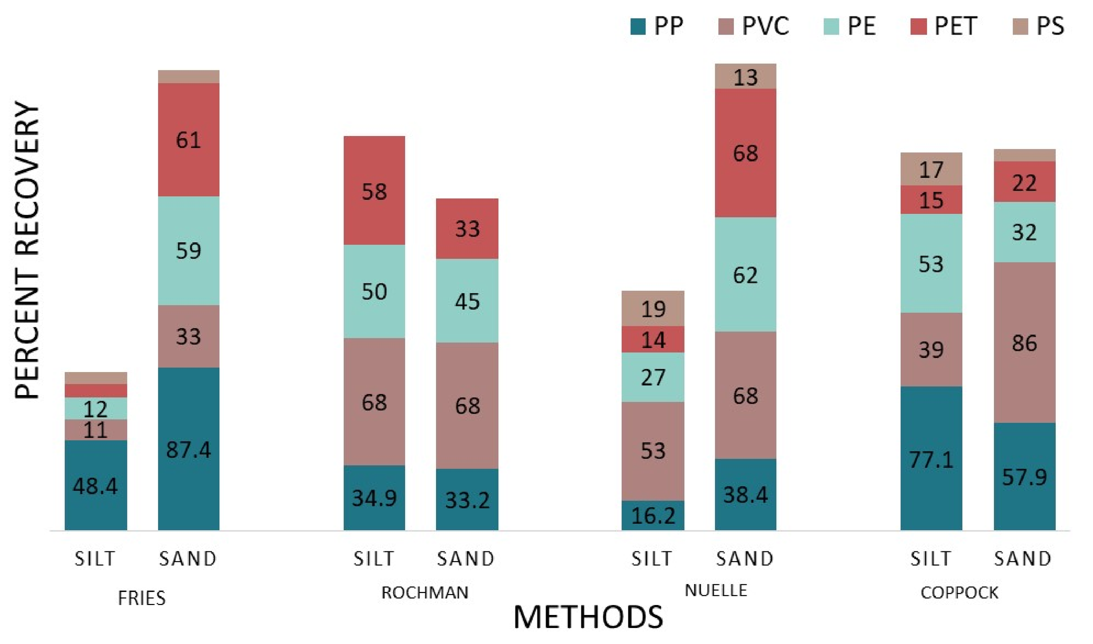

Figure 4. Comparison of microplastic percent recoveries by method.

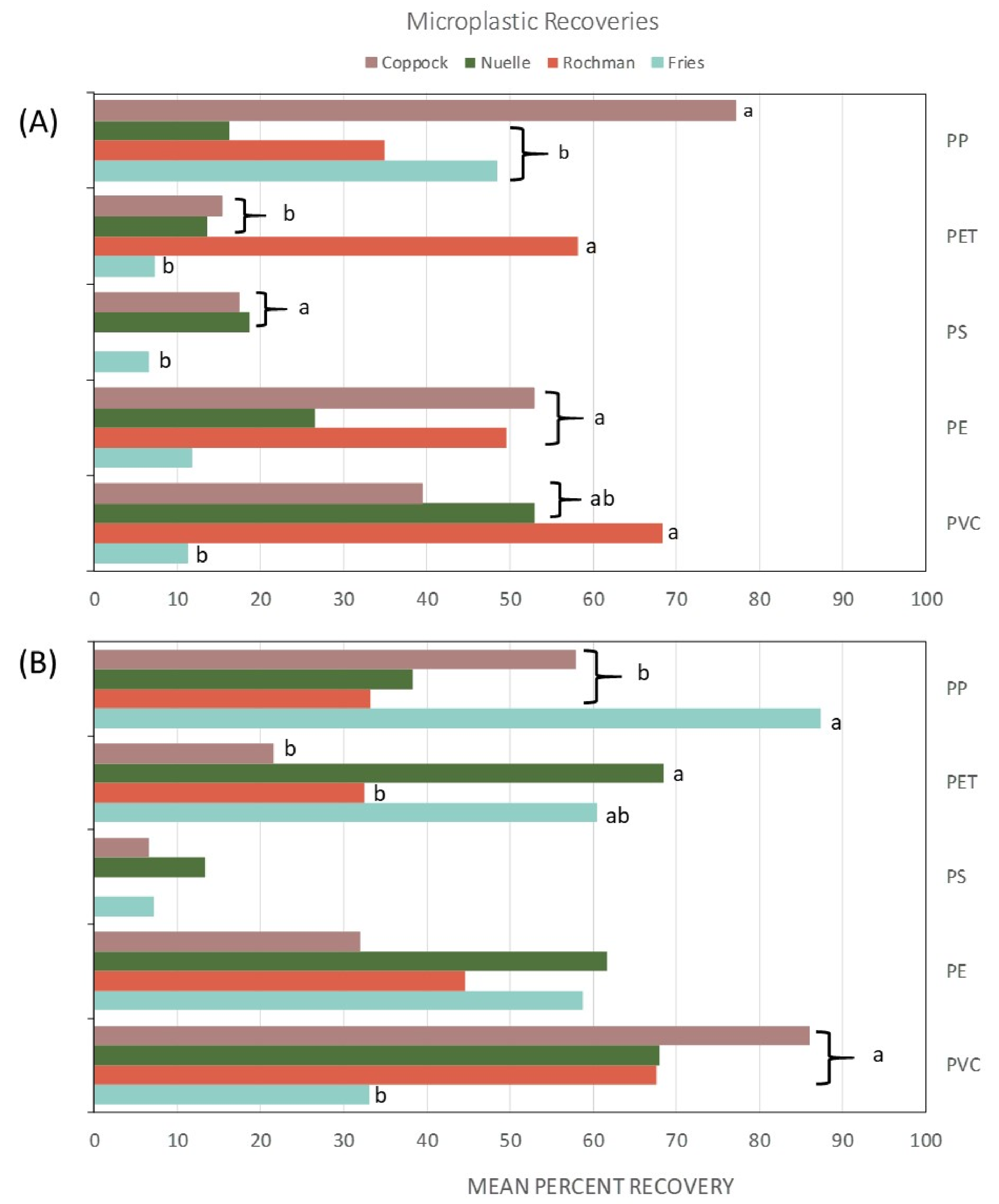

Figure 5. Results of Tukey-Kramer multiple comparisons test when significant differences between methods were detected by analysis of variance (ANOVA) in (A) silty and (B) sandy sediments. Note: Statistical analyses were performed one plastic type at a time.

CONCLUSIONS

- No method successfully recovered >70% of every polymer type.
- Denser plastics often missed by methods with low density salt solutions.
- Visual observation are not an effective technique for identifying plastic particles <50µm.
- Rochman method best at recovering dense plastics from silty sediments.
- Coppock method best at recovering light plastics from silty sediments.
- Nuelle method best at recovering PE, PS, and PET.
- Coppock best at recovering PVC, Fries best at recovering PP from sandy sediments.

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