A small-scale, portable method for extracting microplastics from marine sediments*

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ABSTRACT

Microplastics (plastic particles, 0.1 μm–5 mm in size) are widespread marine pollutants, accumulating in benthic sediments and shorelines the world over. To gain a clearer understanding of microplastic availability to marine life, and the risks they pose to the health of benthic communities, ecological processes and food security, it is important to obtain accurate measures of microplastic abundance in marine sediments. To date, methods for extracting microplastics from marine sediments have been disadvantaged by complexity, expense, low extraction efficiencies and incompatibility with very fine sediments.

Here we present a new, portable method to separate microplastics from sediments of differing types, using the principle of density floatation. The Sediment-Microplastic Isolation (SMI) unit is a custom-built apparatus which consistently extracted microplastics from sediments in a single step, with a mean efficiency of 95.8% (±SE 1.6%; min 70%, max 100%). Zinc chloride, at a density of 1.5 g cm−3, was deemed an effective and relatively inexpensive floatation media, allowing fine sediment to settle whilst simultaneously enabling floatation of dense polymers. The method was validated by artificially spiking sediment with low and high density microplastics, and its environmental relevance was further tested by extracting plastics present in natural sediment samples from sites ranging in sediment type; fine silt/clay (mean size 10.25 ± SD 3.02 μm) to coarse sand (mean size 149.3 ± SD 49.9 μm). The method presented here is cheap, reproducible and is easily portable, lending itself for use in the laboratory and in the field, eg. on board research vessels. By employing this method, accurate estimates of microplastic type, distribution and abundance in natural sediments can be achieved, with the potential to further our understanding of the availability of microplastics to benthic organisms.

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1. Introduction

Microplastics (plastic 0.1 μm–5 mm in size) are ubiquitous throughout the marine environment and are widely regarded as a contaminant of global concern (2008/56/EC Marine Strategy Framework Directive, Descriptor 10, United Nations Sustainable Development Goal 14 target 14.1.1). Over the past 75 years, plastic production has increased dramatically from 1.5 million tonnes to 322 million tonnes per year globally (Plastics Europe, 2016); an estimated 4–12 million tonnes of plastic is predicted to have entered the marine environment from land-based sources in 2010 alone (Jambeck et al., 2014). Microplastic debris is widespread, impinging upon the poles (Obbard et al., 2014), deep sea (Woodall et al., 2014), open ocean (Barnes et al., 2009) and shorelines worldwide (Browne et al., 2011; Nelms et al., 2016). Microplastics are formed in a variety of ways, including: (1) direct manufacture, whereby microscopic or small plastics are purpose made (e.g. cosmetic exfoliates, virgin pre-production pellets); (2) fragmentation of larger pieces of plastic that have degraded after prolonged exposure to the elements (Andrady, 2011); (3) microfibres shed from ropes (Welden and Cowie, 2016) and textiles (Browne et al., 2011; Napper and Thompson, 2016); and (4) tyre and road paint particles transported via run-offs from roads (Boucher and Friot, 2017; Horton et al., 2016).

Owing to their small size, microplastics are bioavailable to a
wide range of organisms. Ingestion has been documented in animals throughout the marine food web, including zooplankton (Desforges et al., 2014), fish (Bellas et al., 2016; Lusher et al., 2013), marine mammals (Lusher et al., 2015; Bravo-Rebolledo et al., 2013), turtles and seabirds (Tourinho et al., 2010). This ingestion of microplastics can negatively affect food intake, reducing the amount of energy available for growth and reproductive success (Cole et al., 2015; Sussarellu et al., 2016; Wright et al., 2013). Plastics can act as a source of chemical contamination, containing plasticizers and additives incorporated into the plastic during manufacture. They may also be vectors for chemicals sorbed onto their surface from the marine environment (Teuten et al., 2009). Plastic debris has been shown to concentrate harmful pollutants up to one million times higher than that of the surrounding seawater (Mato et al., 2001) and consumption of this polluted plastic may result in detrimental effects to marine life (Koelmans et al., 2016).

Benthic sediments have been identified as a potentially important sink for microplastics (Clark et al., 2016; Woodall et al., 2014; Zalasiewicz et al., 2016). Highly impacted coastal sediments can contain up to 3% microplastics by weight (Carson et al., 2011), and Woodall et al. (2014) conservatively estimates that 4 billion fibres km−2 are littering Indian Ocean seamounts and sediments. Environmental models (Lusher et al., 2015 and references therein) have reported the presence of a wide range of microplastic polymer types in sediments, including typically buoyant polymers. Biofouling (Lobelle and Cunliffe, 2011), metal adsorption (Corcoran et al., 2013), and incorporation of microplastics into faecal pellets (Cole et al., 2015; Sussarellu et al., 2016; Wright et al., 2013). Plastics can act as a source of chemical contamination, containing plasticizers and additives incorporated into the plastic during manufacture. They may also be vectors for chemicals sorbed onto their surface from the marine environment (Teuten et al., 2009). Plastic debris has been shown to concentrate harmful pollutants up to one million times higher than that of the surrounding seawater (Mato et al., 2001) and consumption of this polluted plastic may result in detrimental effects to marine life (Koelmans et al., 2016).

2. Methods

2.1. Floatation media

Solutions of sodium chloride (NaCl), sodium iodide (NaI) and zinc chloride (ZnCl2) were prepared by dissolving the salts in ultrapure water to achieve densities in the range 1.2–1.8 g cm−3 (Table 3); solutions were filtered (10 μm Whatman nucleopore membrane) to remove any contaminants prior to use. The financial cost of preparation of media was calculated by averaging the cost of salts from three scientific suppliers (i.e. Fisher Scientific, Sigma Aldrich and APC Pure; December 2016) and adjusted for the preparation of solutions (amount added to 1 L ultra-pure water) at the appropriate density (Table S12; Supplementary Information).

2.2. Sediment-Microplastic Isolation (SMI) unit

In evaluating existing microplastic extraction protocols (Table 1), we identified the need for a method that allows rapid, simple and efficient extraction of microplastics from a range of sediment types. We set out to design a compact extraction unit that can be easily decanted in a single step and quickly cleaned to avoid cross-contamination. Following optimisation, we constructed the Sediment-Microplastic Isolation (SMI) unit (Fig. 1). The unit was constructed using 63 mm PVC piping and ball valve, secured to a PVC plate with PVC welding rod for stability (see Table S11 for material information and costs) with dimensions of 130 (w) x 130 (d) x 380 mm (h), and a weight (excluding floatation media) of 1.5 kg. The unit was designed so that all internal sides were smooth with no protruding surfaces, allowing free movement of the particles, thus avoiding any microplastics becoming trapped within the unit.

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2.3. Cleaning, purging and priming the SMI

All SMI components were thoroughly rinsed with ultrapure water prior to assembly; particular attention was given to cleaning the ball valve owing to its relative complexity. Following assembly, 700 mL of filtered ZnCl₂ solution (1.5 g cm⁻³) was poured into the SMI unit, ensuring the ball valve was completely submerged. The ball valve was set in the open position and the ZnCl₂ solution was allowed to escape of trapped air bubbles. The unit was left to settle until the supernatant was clear of sediment. Next, the valve was carefully closed and the supernatant in the headspace was vacuum filtered (Millipore) through a 30 μm nylon mesh (or split over multiple meshes if high quantities of organic material present), retaining the zinc chloride for further use. The headspace was rinsed thoroughly with ultrapure water to recover any remaining particles. Meshes were transferred to a clean Petri dish and sealed with Parafilm, pending examination under a microscope. After each extraction,
the SMI was cleaned with ultra-pure water and purged again before processing the next sample. Procedural blanks (ZnCl₂ excluding sediment) were carried out prior to first use and after every three samples as a contamination control measure.

2.5. Extraction of microplastic from artificially spiked sediments — SMI validation

To evaluate the extraction efficiencies of the SMI unit, we used natural and untreated fine sediment (Table 2) spiked with known concentrations of microplastics. Sediment samples were sourced from the entrance to the Plym estuary, Plymouth Sound, UK (N 50°21.717′; W 4°08.055′) using a benthic multicorner (four Perspex cores measuring 50 cm long x 10 cm diameter). Samples were dried at 50 °C for approximately 72 h then stored in a clean polyethylene bag (Sigma Aldrich Z162965). Artificially incorporated microplastics (Table S13) included: (1) weathered polyethylene filaments (200–1000 μm), and (2) weathered nylon filaments (200–1000 μm long) both sourced from Cockleridge beach (Devon, UK; N50°28.136′; W03°87.150′) in 2014 and hand-cut to the given sizes using dissecting scissors; (3) virgin polyvinylchloride (100–800 μm, Goodfellow); and (4) manufactured low density polyethylene (400–1000 μm), prepared by milling clean milk bottle lids with a cryogenic-grinder (SPEX Freezer-Mill® 6870) and then cutting to appropriate size using dissection scissors. Spiked plastics were distinctive, both in colour and shape and obviously cut at both ends, ensuring that only spiked plastics were counted in the trials. Microplastics (50 combined particles per replicate) were mixed with 30 g sediment in a clean, ceramic bowl, any solidified sediment was gently broken up using the weight of a pestle. Plastic-spiked sediment samples (n = 5) were then added to the SMI unit with 700 ml ZnCl₂ (1.5 g cm⁻³). The plastics were inspected for signs of degradation post extraction.

2.6. Extraction of microplastic from natural sediment samples — environmental validation

The applicability of the SMI unit in isolating microplastics from natural sediment samples was also verified by testing the procedure on locally sourced sediments of varying grain size. Natural sediment was sourced from three sites in the western English Channel (Table 2). Fine and medium sediments (n = 3; Table 2) were sampled using a benthic multicorner deployed from the RV Plymouth Quest at sites local to Plymouth; the top 2 cm of each core was used for microplastic extraction. Tide time was not controlled for due to logistic constraints. Coarse sand (n = 3) was sampled using a clean stainless steel measuring cup from the intertidal zone at the cliff base at Portwrinkle beach, Cornwall during low tide. All samples were immediately transferred to a clean foil tray and sealed. Sediment was dried at 50 °C for approximately 72 h then microplastics extracted using the SMI unit as previously described (up to 50 g dry sediment per extraction). Once complete, nylon meshes were visually examined under a microscope (Leica,×25 magnification) for particles with a synthetic appearance; i.e. lacking cell structure, unnatural appearance in shape, colour or texture (Lusher et al., 2016). Isolated microplastics were photographed and characterised by quantifying the shape (fragment, fibre or nurdle), colour and size of each particle. Particles were chemically quantified by Fourier Transform Infrared spectroscopy (Agilent Cary 630 and Bruker Vertex 70 with Hyperion 1000 microscope). Data were normalised by the dry weight (g) of sediment added to the SMI for extraction.

2.7. Contamination control

Contamination controls and procedural blanks were implemented during field sampling and sample processing, per the protocols of Lusher et al., 2016. All equipment was rinsed first with tap water, then twice with ultra-pure water before covering with clean foil. A dampened glass fibre filter (GF/C) paper was left open to the air both on board RV Quest and in the laboratory at each stage of processing and screened for plastic contamination using a light microscope (Leica,×25 magnification). Procedural blanks were used throughout to control for equipment contamination and samples were processed inside a clean laminar flow cabinet. Bench tops and microscope were cleaned prior to picking microplastics.
from filtered samples, and care was taken to expose samples for minimal periods. At times when using the laminar flow cabinet was not appropriate, a clean polyethylene cape was created around the microscope (Fig. S1; Supplementary Information). A cotton laboratory coat was worn at all times.

3. Results and discussion

3.1. Floatation media

A range of densities of three different salt solutions were trialled to determine the optimal conditions to float microplastic particles from sediment samples, balancing the attainability of high-density solutions and financial cost (Table 3). Sodium chloride proved the cheapest option (£4.17 L⁻¹; referred to as ‘1 cost unit’ for comparative purposes; see Table 3.), however the maximal achievable density is just 1.2 g cm⁻³. Numerous field studies have reported microplastic concentrations following extraction using NaCl. These include high profile studies by Browne et al. (2011), who highlight that coastlines are contaminated with microplastic particles on a global scale and positively correlated with densely populated areas, and Woodall et al. (2014), who identified that microfibres are prevalent in deep sea sediments in abundances of up to four orders of magnitude higher than that of contaminated surface waters. However, while saturated NaCl is adequate in extracting low density plastics from sediments, it precludes denser plastics such as PVC (1.3–1.45 g cm⁻³) and polyethylene terephthalate (PET, 1.38 g cm⁻³), commonly used in textiles and to produce plastic bottles, from being lifted. Therefore, whilst NaCl is a cheap, inert option to use in microplastic studies, its use could result in an underestimation of the abundance of plastics found, particularly high density plastics. Sodium iodide can be prepared to higher underestimation of the abundance of plastics found, particularly high densities (1.6 g cm⁻³), the SMI unit is straightforward in design and use, relatively cheap to produce, with each unit costing around GBP £50 (excl. VAT; Table S11). This allows for multiple units to be manufactured and used simultaneously, increasing the scope for sample replication, and reducing the time required to process all samples. Its design mirrors that of the Munich Plastic Sediment Separator [MPSS – (Imhof et al., 2012)], whereby sediment is mixed at the base of a vessel, and density floatation is used to float plastics above a shut-off valve. The MPSS is designed to extract plastics from up to 6 kg of sediment using 12 L of dense salt media, with aeration to adjust the relative density. As such, the MPSS is constructed entirely of stainless steel, stands at approximately 1.75 m tall by 36 cm wide and includes a base equipped with an electric motor to stir the sediment. While the MPSS is well suited for isolating microplastics from large volumes of sediment, the expense and complexities of manufacturing, size, weight and volume of floatation media required, limit its functionality and feasibility when processing numerous replicates of small samples.

4. SMI validation

4.1. Results from artificially spiked sediments

Microplastics artificially incorporated into fine estuarine sediments were extracted using the SMI unit with ZnCl₂ at a density of 1.5 g cm⁻³. Mean extraction efficiencies, based on fibrous and particulate microplastics of different densities retrieved in a single step, ranged from 92 to 98% (n = 5, mean 95.8 ± SE 1.6; Fig. 2.) and were comparable with those of the MPSS (Imhof et al. (2012)), for which a mean 95.5% recovery rate for <1 mm microplastics was identified. No degradation of spiked plastics was observed after immersion in ZnCl₂ for 24 h.

Losses in microplastic recovery were found to arise if the unit

![Fig. 2. Mean (±SE 1.6; min 70%; max 100%) percentage recovery of microplastics (n = 10–20) from artificially spiked sediment (n = 5).](image-url)
was not primed with the floatation media prior to adding the sample. Indeed, if the space inside the ball valve is not filled with fluid, the media will be agitated when the valve is opened as the liquid floods the cavity, potentially leading to loss of plastics otherwise retrieved within the headspace of the SMI. Other potential losses may occur if very small microplastics become trapped within the sediment as it sinks back down to the bottom of the unit. It is therefore important to ensure the unit is not overfilled with sediment, thus avoiding a sub-optimal ratio of sediment to floatation media, recommended here up to 50 ml sediment to 700 ml media. Similarly, it is also recommended that the sediment is briefly mixed again once the sediment begins to settle, to avoid microplastics becoming trapped within air bubbles in the sediment. Some key benefits of using the SMI unit in conjunction with ZnCl$_2$ (1.5 g cm$^{-3}$) over other microplastic extraction methods (see Table 1) are: the combination of high extraction efficiency in a single step, simplicity, affordability and a compatibility with all sediment types.

The classic decanting method (Hidalgo-ruz et al., 2012; Thompson et al., 2004), though simple in design, has a relatively lower recovery rate (35% pers. obs. 40% Imhof et al., 2012), due to plastics adhering to the inside of the vessel as the media is decanted. To combat this low extraction efficiency, the technique is often repeated 3–5 times, extending the processing period for each sample (Claessens et al., 2013; Fries et al., 2013; Nuelle et al., 2014). Studies employing this method may therefore underestimate the...
number of microplastics. Conversely, extending the sample processing time may increase the risk of external contamination. We propose, that in using the SMI unit, the user has the advantage of being able to rinse the entire headspace multiple times without resuspending the settled sediment, therefore reducing the need for repetitive processing and limiting opportunities for external contamination. The SMI has also proven compatible with finer sediments (e.g. estuarine silt). This contrasts with a number of existing methods (e.g. the elutriation and aeration chamber presented by Caesens et al., 2013), which works well with coarse sand but was found, when replicated for use in early trials, to clog irreprovably when using the fine, muddy estuarine sediments (pers. comms. Dr Andy Watts, University of Exeter).

4.2. Results from environmental samples

The SMI protocol extracted microplastic debris from all environmental samples, including coarse beach sediments and fine estuarine mud. Microplastic concentrations and type varied across samples and sites, ranging from 29.3 to 144.1 synthetic particles kg$^{-1}$ dry sediment (mean $\pm$ SE: 67.4 $\pm$ 13.2) across the sites sampled (Fig. 3). In the coarse sediments, 66.7 particles kg$^{-1}$ (mean $\pm$ SE 17.6) were unidentified. Microplastics consisted of nurdles, fragments and fibres in a variety of colours, including blue, green, orange and mauve ranging in size from 100 $\mu$m to 10 mm in length and 30 $\mu$m to 4.3 cm wide, with mean dimensions of 3325 $\mu$m $\times$ 2117 $\mu$m. Polyethylene and ethylene copolymers were the dominant constituents of the microplastics found in the coarse sand (67%). These polymers are the most widely manufactured plastic type, commonly used in packaging. Polypropylene (8%), frequently used to make ropes, styrene (8%) and unidentified particles (17%) were also present. Fine sediment yielded 72.2 particles kg$^{-1}$ (mean $\pm$ SE: 36.2), all were fibrous and ranging in length from 80 $\mu$m to 5000 $\mu$m, 20 $\mu$m–40 $\mu$m wide and blue, red, black or transparent in colour. Semi-synthetic rayon, commonly used in textiles and sanitary products, was the predominant polymer type (67%), with polyester (13%), polyethylene terephthalate (PET - 7%), which is the main polymer used in plastic bottle production, polypropylene (7%) and unidentified particles of synthetic appearance (7%) present. Medium sediment yielded 63.3 synthetic particles kg$^{-1}$ (mean $\pm$ SE: 21.5) and were predominantly fibres, with one fragment present. The particles were red, grey, blue, transparent or green in colour and ranged from 400 $\mu$m to 5000 $\mu$m in length and 30 $\mu$m–200 $\mu$m wide. Polyester (25%), a common polymer in the manufacture of clothing, and acrylic (25%), frequently used in optical applications and additives in paints, were the most abundant. Also present were ethylene propylene (12.5%), rubber widely used for its insulation properties, polypropylene (12.5%), rayon (12.5%), and unidentified particles (12.5%) (Fig. 3).

Whilst this method has proven reliable in microplastic extraction from a range of sediment types, it is ultimately reliant on the user to manually sort and extract the plastics which is labour intensive and may introduce potential bias. Longer term, a shift to a more automated method of analysis is envisaged; however the infrastructure and technology are not currently available.

5. Conclusions

A clear understanding of the microplastic availability within marine sediments requires accurate data on microplastic abundance in natural systems, of which there is a paucity at present. Despite calls for consistently applied sampling and extraction strategies, this is currently still lacking. Here we have presented a method to extract microplastics from sediments using a specially constructed Sediment–Microplastic Isolation (SMI) unit, in combination with zinc chloride solution (1.5 g cm$^{-3}$), able to extract microplastics from sediments with a mean recovery rate of 95.8% in a single step. The method is cost effective, encouraging universal use regardless of budget, thereby promoting harmonised sampling and working towards achieving comparable data sets across studies. The protocol is applicable to a range of sediment types, with microplastics successfully isolated from estuarine silts and clay, and coarse beach sand. Zinc chloride was determined the most appropriate media tested for floatation of microplastics, achieving high densities with relatively low expense (GBP £35 L$^{-1}$ at 1.5 g cm$^{-3}$). An optimal density of 1.5 g cm$^{-3}$ was determined, balancing the requirement for media dense enough to allow floatation of different polymer types whilst allowing fine sediments to settle out of suspension to achieve the desired separation. The small dimensions and low weight lend the SMI for use in multiple settings, including laboratories and field based work such as onboard research vessels. Without accurate data on the field occurrence of microplastics in marine sediments we cannot regulate this widespread pollution of the marine environment and food web. A method to fill important data gaps regarding the availability of microplastics to benthic organisms is described here and made available.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.envpol.2017.07.017.

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