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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.

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

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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 seamount sediments. Environmental studies (Lusher, 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), mineral adsorption (Corcoran et al., 2015) and incorporation of microplastics into faecal pellets (Cole et al., 2016) and marine aggregates (Long et al., 2015) can decrease the buoyancy of plastics, facilitating their movement to the seafloor. Within the sediment, microplastics may therefore become bioavailable to a wide range of benthic fauna, including commercially important species, such as Norway lobster (Murray and Cowie, 2011) and shellfish (Rochman et al., 2015) that contribute to biochemical and nutrient cycling processes (Queirós et al., 2015; Zhang et al., 2015). Following exposure to polyvinylchloride (PVC) microplastics, ecologically important intertidal polychaete lugworms, *Arenicola marina,* suffered a 50% reduction in energy reserves (Wright et al., 2013), increased metabolic rates and reduced bioturbation (Green et al., 2016) with impacts on its role in ecosystem process mediation (Volkenborn et al., 2007).

To gain a clearer understanding of microplastic availability to marine life, and thus of risks posed to the health of benthic communities and associated ecological processes, it is important to obtain accurate measures of microplastic abundance in sediments. Indeed, a recent review highlighted the difficulties in developing a global picture of benthic microplastic prevalence due to the lack of reliable microplastic abundance measurements (Underwood et al., 2017). This is largely due to the costs, impracticalities or inefficiencies associated with existing methods. We therefore need to promote harmonised, practical and representative sampling, sample preparation and microplastic detection (Horton et al., 2017; Van Cauwenberghe et al., 2015). The principle of density floatation is commonly employed to separate less dense plastic polymers from denser sediment particles, and a range of high-density salt solutions have been used to extract microplastics from marine sediments (Hanvey et al., 2016; Horton et al., 2016; Thompson et al., 2004 and references within Table 1. - this table is not intended to be exhaustive but highlights other floatation methods commonly cited in the literature). However, such methods have been disadvantaged with a number of drawbacks, including complexity (Claessens et al., 2013), expense (Imhof et al., 2012), low extraction efficiencies (Hidalgo-ruz et al., 2012; Imhof et al., 2012), incompatibility with very fine sediments (Claessens et al., 2013; Fries et al., 2013), particle degradation from floatation media (Lusher et al., 2016), and expense of consumables, e.g. Metatungstate solution in the NOAA approved protocol (Masura et al., 2015). The decanting of floating plastic whilst simultaneously avoiding disruption of the settled sediment poses a challenge, typically yielding low extraction efficiencies and hence requiring repeat extractions (Hidalgo-ruz et al., 2012; Imhof et al., 2012). Other methods require several steps to retrieve microplastics (Claessens et al., 2013; Fries et al., 2013; Nuelle et al., 2014; Stolte et al., 2015) and may include equipment that suit extraction from coarse sediments such as an elutriation step (Claessens et al., 2013) or use of a separation funnel (Fries et al., 2013), but clog when using very fine sediments (pers. comms. Dr. Andy Watts, University of Exeter and pers. obs.). The Munich Plastic Sediment Separator [MPSS - (Imhof et al., 2012)] isolates microplastics above a shut-off valve and achieves recovery rates of 95.5% (microplastics < 1 mm). However, the MPSS was designed for use with large quantities of sediment (6 kg) and is fabricated from stainless steel standing at approximately 1.75 m tall, thereby expensive to produce and limiting its portability and feasibility when processing numerous replicates of small samples.

Here, we describe the construction and application of a small-scale, portable microplastic extraction unit that mirrors the design of the MPSS, and compare the viability and financial cost of three high-density salt solutions: sodium chloride (NaCl), sodium iodide (NaI) and zinc chloride ($ZnCl_2$), tested for use with the unit. We test the efficiency of the unit by artificially spiking sediment with known quantities of microplastics (polyethylene, polyvinyl chloride and nylon) and validate its use with environmental samples of varying type. We present an optimised method that is applicable for use with a range of sediment types, suits most budgets and which can be used both in the field and the laboratory to isolate microplastics from benthic samples.

2. Methods

2.1. Floatation media

Solutions of sodium chloride (NaCl), sodium iodide (NaI) and zinc chloride ($ZnCl_2$) 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 (GBP L^{-1}) 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 SI2; 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 SI1 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.

Table 1
Examples of existing floatation methods commonly used to extract microplastics from sediments.

Floatation extraction technique	Floatation media	Repetitions	No. steps	Sediment type	Efficiency	Size	References
Decanting	NaCl; NaI	2–5	1	Fine (estuary) Coarse (beach)	35% (pers. obs.)	<1 mm	(Hidalgo-ruz et al., 2012; personal observations)
Modified decanting	NaCl; NaI; $ZnCl_2$	1–2	1–2	Fine (river; mangrove)	99%; 40–72%	1 - 5 mm <1 mm	(Imhof et al., 2012; Mohamed Nor and Obbard, 2014)
Elutriation, aeration & centrifuge	H_20; NaCl; NaI;	1 × NaCl elutriation 3 × NaI centrifuge	2	Coarse (sand)	97 - 98%	<1 mm	(Claessens et al., 2013; Wessel et al., 2016)
Aeration & ball valve (MPSS)	$ZnCl_2$	1	1	Fine (river)	100% 96%	1 - 5 mm <1 mm	(Imhof et al., 2012)
Froth floatation	Pine oil, froth conditioner, dishwasher tablet	1	1	Fine (river)	55%	1 - 5 mm	(Imhof et al., 2012)
Air-induced overflow, oxidation & decanting	NaCl; NaI	5	3	Coarse (beach)	68 - 99%	1 mm	(Nuelle et al., 2014)
Separation funnel	NaCl	2	2	Coarse (beach)	80–100%	1 mm	(Fries et al., 2013)
Pipetting & decanting	$CaCl_2$	1	2	Coarse (beach)	55%	<1 mm	(Stolte et al., 2015)
Overflow	NaI; NaCl	3	1	Fine (sound)	48%	<1 mm	(Personal observations)

Table 2
Description of sediments used for environmental testing of SMI unit.

Site	Lat. long.	Descriptor	Type	Grain size ($\bar{X}$ μm)	*n*	Sampling date
Plym Estuary	N 50°21′716″ W 4°08′073″	Fine	Clay/silt	10.25 (±SD 3.02)	3	23.06.16
Plymouth Sound Breakwater	N 50°20′174″ W 4°08′605″	Medium	Silt	20.78 (±SD 3.05)	3	23.06.16
Portwrinkle Beach	N 50°21′390″ W 4°18′22.9″	Coarse	Coarse sand	149 (±SD 49.86)	3	13.12.16

Table 3
Comparative amount of salts (g) added to 1 L ultra-pure water to achieve specific densities, and their associated costs.

Salt	Density (g cm^{-3})	Amount added to 1 L H_20 (g)	Cost (GBP L^{-1})	Relative cost unit
Sodium chloride (NaCl)	1.2	337	£ 4.17	1
Sodium Iodide (NaI)	1.3	494	£ 85.44	20.5
	1.5	1000	£ 172.95	41.5
Zinc chloride ($ZnCl_2$)	1.3	500	£ 18.06	4.3
	1.5	972	£ 35.10	8.4
	1.8	1800	£ 65.00	15.6

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 $ZnCl_2$ solution (1.5 g cm^{-3}) was poured into the SMI unit, ensuring the ball valve was completely submerged. The ball valve was primed by opening and closing several times, making sure the internal cavity was filled so as to avoid agitation upon valve closure during sample processing. The solution was topped back up to approximately 90 mm above the open valve (approximately 50 mL) and the unit left for 5 min to allow any externally-derived contaminants to float to the surface. After 5 min, the valve was set in the open position and the $ZnCl_2$ solution filtered through a 25 μm nylon mesh into a clean flask for continued use, rotating the unit to ensure all internal sides were clear of contamination. This step was undertaken prior to each extraction and took no more than 10 min.

2.4. Microplastic extraction from sediment

During extraction from sediment samples, all cleaned equipment was placed inside a laminar flow hood and covered with clean aluminium foil to minimise contamination. On each occasion, a dry (30–50 g) sample, clean magnetic stir bar and 700 mL of $ZnCl_2$ were added to the purged SMI unit. A magnetic stirring plate was used to mix the sediment for 5 min, and then the sediment allowed to settle for 5 min, followed by 3 short stirring pulses to allow the 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 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,

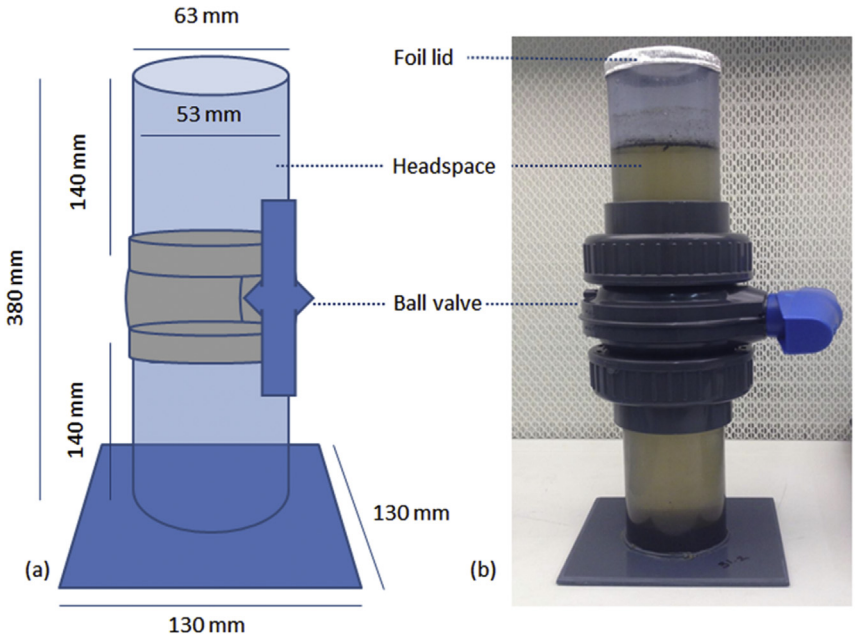

Fig. 1. Schematic (a) and photograph (b) of Sediment-Microplastic Isolation (SMI) unit. Photograph depicts SMI unit within contamination control cabinet with ball valve in closed position, denser sediment settled at the bottom of $ZnCl_2$ solution (1.5 g cm^{-3}) and less dense particles floating on top.

the SMI was cleaned with ultra-pure water and purged again before processing the next sample. Procedural blanks ($ZnCl_2$ 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 multicorer (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 SI3.) 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 $ZnCl_2$ (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 multicorer 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. SI1; 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 densities than sodium chloride, however achieving a density of 1.5 g cm^{-3} proved the most expensive option at £172.95 L^{-1} (20.5 cost units) and therefore was consequently eliminated from our trials. Where NaI has been used to extract microplastics, multi-step methods are necessitated to minimise the volume of NaI required (Claessens et al., 2013; Nuelle et al., 2014, Table 1). Zinc chloride as a floatation medium has the benefit of attaining densities >2 g cm^{-3} and is relatively inexpensive at £35.10 L^{-1} (8.5 cost units) to prepare a density of 1.5 g cm^{-3}, enabling its use at greater volumes at higher densities. As such, $ZnCl_2$ has been used to quantify microplastic abundance in a number of microplastic studies (Horton et al., 2016; Imhof et al., 2012; Liebezeit and Dubaish, 2012). In this study, at very high densities (1.6–1.8 g cm^{-3}), the fine sediment used for SMI method validation remained in suspension, making it impractical to use for plastic extraction. Therefore, considering the relative achievable density of NaCl and the expense of NaI, from our results, $ZnCl_2$ was deemed the most appropriate salt solution for floatation of microplastics using the SMI unit, at an optimal density of 1.5 g cm^{-3} when extracting from fine sediment. This density, whilst it precludes aggregates or composites denser than 1.5 g cm^{-3}, balances the requirement for the sediment to settle, whilst still dense enough to enable floatation of denser plastics such as PVC and PET.

3.2. Sediment-Microplastic Isolation (SMI) unit

The Sediment-Microplastic Isolation (SMI) unit is a compact, portable device that extracts microplastics from different sediment types in a single step, with reproducible results. A prototype of the SMI was constructed from glass and steel, however we identified that $ZnCl_2$ reacted with the steel. The advantages of manufacturing the SMI using PVC include resistance to corrosion, plus ease of construction, reduced costs, durability and weight. A quotation was obtained to construct a version of the SMI from stainless steel, however at GBP £640 (excl. VAT) per unit, it was no longer a cheap option, therefore potentially hindering the harmonisation of microplastic extraction methodology across studies. Constructing the unit from plastic does have a potential downside; long term use has not been tested in this study, and over time there is potential the continued use of $ZnCl_2$ could result in the degradation (e.g. fracturing, cracking) of the SMI unit. With this in mind, if regular inspection and procedural blanks reveal contamination, the unit should be replaced, which is made feasible by the low cost of the unit. In following the prescribed purging method, the SMI unit extracted microplastics from different sediments whilst avoiding self-contamination (corroborated by procedural blanks); the unit can be dismantled for easy, thorough cleaning between samples. The SMI unit is straightforward in design and use, relatively cheap to produce, with each unit costing around GBP £50 (excl. VAT; Table SI1). 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 $ZnCl_2$ 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 $ZnCl_2$ for 24 h.

Losses in microplastic recovery were found to arise if the unit

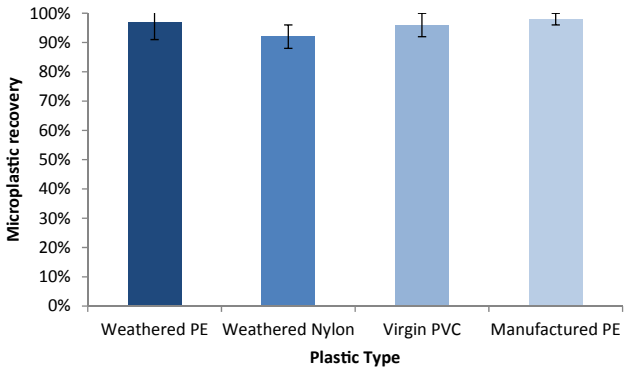

Fig. 2. Mean (±SE 1.6; min 70%; max 100%) percentage recovery of microplastics ($n = 10–20$) from artificially spiked sediment ($n = 5$).

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

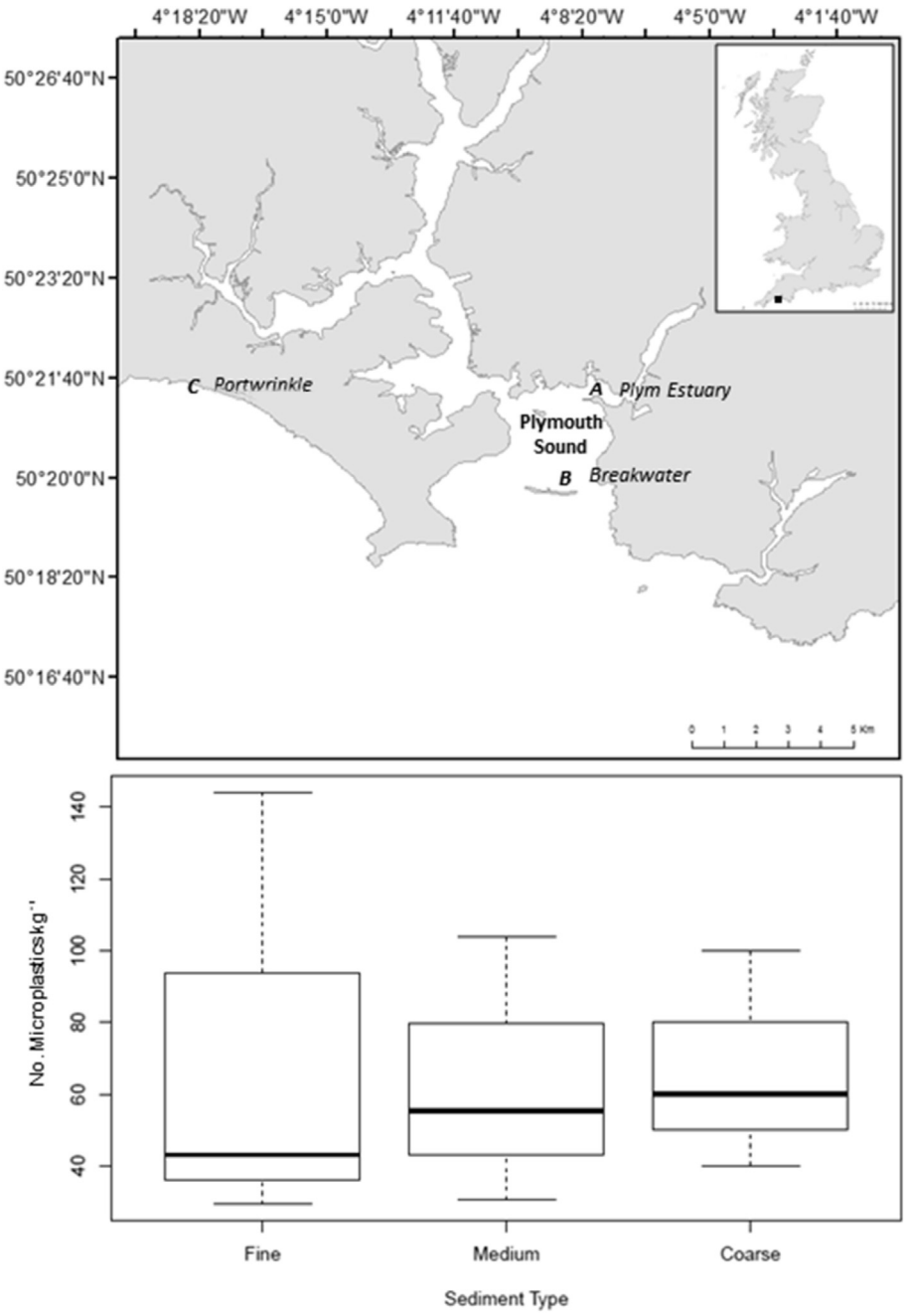

Fig. 3. Sample collection sites for SMI validation, for fine (A), medium (B) and coarse (C) (unspiked) sediment (Table 2). Box and whisker plots show the median, interquartile and full range of microplastics extracted from each sediment type.

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 re-suspending 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 Claessens et al., 2013), which works well with coarse sand but was found, when replicated for use in early trials, to clog irrevocably 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 ± SE: 67.4 ± 13.2) across the sites sampled (Fig. 3.). In the coarse sediments, 66.7 particles kg^{-1} (mean ± SE 17.6) were identified. Microplastics consisted of nurdles, fragments and fibres in a variety of colours, including blue, green, orange and mauve ranging in size from 100 μm to 10 mm in length and 30 μm to 4.3 cm wide, with mean dimensions of 3325 μm × 2117 μm. 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 ± SE: 36.2), all were fibrous and ranging in length from 80 μm to 5000 μm, 20 μm–40 μm 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 ± 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 μm to 5000 μm in length and 30 μm–200 μm 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 on-board 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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