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Microplastics in Galway Bay: A comparison of sampling and separation methods

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ABSTRACT

Microplastics, an emerging pollutant, are recognised as having a ubiquitous distribution in the environment. Currently several benthic sampling tools are being employed to collect subtidal marine sediment, however, there are no comparative studies on the efficiency of these tools to sample for microplastics or the subsequent extraction methods of microplastics from these marine sediments. This study addresses these knowledge gaps by comparing commonly applied benthic sampling tools (Van Veen grab, box corer, gravity corer) and a variety of density separation methods (elutriation column, sodium chloride solution, sodium tungstate dihydrate solution) for microplastic collection and processing.

Each sampling tool was tested at the same station and the collected sediment was used to assess the extraction performance for the different density separation techniques. No statistically significant differences were found between the concentrations of microplastics extracted for any of the sampling tools. However, there were significant differences between the density separation methods using sodium tungstate dihydrate and sodium chloride solution and the elutriation method. This preliminary study provides evidence that the sampling tools tested are both suitable and proficient at determining the abundance of microplastics in sediments. Sodium tungstate dihydrate proved to be a novel and feasible option for dense liquid separation of microplastics in subtidal marine sediments. These results will allow for more confidence in data quality when comparing future surveys applying different benthic sampling tools.

1. Introduction

Microplastics, an important part of marine anthropogenic litter, are included within the United Nations (UN) Sustainable Development *Goal 14: Life Below Water* under the auspices of marine pollution; and are now thought to be ubiquitous having been recorded from surface waters to deep sea sediments (Thompson et al., 2004; Moore, 2008; Doyle et al., 2011; Eriksen et al., 2013; Hidalgo-Ruz et al., 2013; Eriksen et al., 2014; Dean et al., 2015; Enders et al., 2015; Taylor et al., 2016). The UN's framework aims to globally prevent and significantly reduce marine pollution by 2025, particularly pollution resulting from land-based activities. In a European context, the Marine Strategy Framework Directive (MSFD) has set a 2020 target to achieve Good Environmental Status (GES) using a set of descriptors which include marine litter and microplastics (Descriptor 10).

Plastics were first reported in coastal waters in the 1970s (Carpenter et al., 1972), while, the term microplastics was first brought into the marine anthropogenic litter vernacular in 2004 by Thompson et al. (2004), but it is still difficult to get a consensus on the definition. The National Oceanic and Atmospheric Administration (NOAA) suggested that the term "microplastic" be applied to all particles < 5 mm (Arthur et al., 2009). However, the United Nation Environment Programme (UNEP) and Global Experts on the Scientific Aspects of Marine Environmental Protection (GESAMP) guidelines, consider microplastics to be particles between 1 mm and 1 μm and identify nanoplastics as particles < 1 μm (GESAMP, 2015a; UNEP, 2016).

All microplastics can be classified as either primary or secondary (Cole et al., 2014). Primary microplastics have been manufactured to be of microscopic dimensions for example facial cleanser exfoliants (often termed microbeads). Secondary microplastics are the result of fragmentation from a larger plastic material for example materials discarded from fishing vessels or fibres from synthetic textiles (Duis and Coors, 2016; GESAMP, 2015b; Koelmans et al., 2014; Napper and Thompson, 2016).Microplastics can be categorised by their morphology into several categories including spheres, pellets, fragments or fibres (Wright et al., 2013; Frias et al., 2018).

There are a number of benthic sampling tools which are regularly employed for the collection sediment samples (Eleftheriou and

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Mcintyre, 2005). The choice of tool largely depends on logistics, the quality of sediment required, the sediment type on site and/or the objective of the survey, for example, contaminant studies may be looking for depth of penetration and require a core to be taken (Mudroch and Azcue, 1995). Because there are often discrepancies in how much metadata is reported, particularly in relation to the sediment type and potential sources of microplastics, comparability between studies is quite difficult (Hidalgo-Ruz et al., 2013; Rocha-Santos and Duarte, 2015; Van Cauwenberghe et al., 2015). Beaches are frequently assessed for microplastics due of their financial and logistical accessibility over projects that require ship time. Not only can beaches be easily surveyed, but also larger sample sizes can be taken with the aid of citizen science and coordinated by NGOs and researchers alike (Mouat et al., 2010; Smith and Edgar, 2014; Newman et al., 2015; Hidalgo-Ruz et al., 2013). As research moves into the subtidal ecosystem, divers can be supplied with cores to collect samples from shallower waters (Eleftheriou and Mcintyre, 2005). Tools usually used for sampling fauna in sediments can dually be used to sample microplastics in sediment. Benthic grabs have been used to collect sediments in many subtidal microplastic studies (Thompson et al., 2004; Claessens et al., 2011; Van Cauwenberghe et al., 2013a; Fischer et al., 2015). Subtidal benthic sampling tools include multicorers (Van Cauwenberghe et al., 2013b), megacorers (Woodall et al., 2015b), sleds, grabs, box corers, while macroplastic collection tools include ROVs, dredges, and trawls (Koutsodendris et al., 2008; Rodríguez and Pham, 2017).

Once samples have been collected, there are a multitude of methods currently being employed to extract, process, and analyse the quantity and type of microplastics present (Pham et al., 2014; Masura et al., 2015; Song et al., 2015; Shim et al., 2016; Karami et al., 2017). The procedure for seawater samples generally involves a two-step process i.e. sieving followed by filtering, however, in some cases the secondary removal process of filtering is not included (Hidalgo-Ruz et al., 2013). Processing sediments, however, can vary from a one step process of sieving to several different two-step processes to separate the microplastics from the original matrix (Hidalgo-Ruz et al., 2013).

Density separation is the most widely reported method for the extraction of microplastics from marine sediments by floatation and subsequent filtration of the supernatant (Quinn et al., 2016). Although sea water (Kusui and Noda, 2003) and distilled water (Alomar et al., 2016) have been used for density separations, the most frequently used dense liquid separation method is sodium chloride (NaCl) (Thompson et al., 2004). There are denser solutions to extract heavier plastics (polyvinyl chloride (PVC), polyethylene terephthalate (PET), nylon (PA) and polytetrafluoroethylene (PTFE)) like sodium iodide (NaI) (Claessens et al., 2013; Dekiff et al., 2014), sodium polytungstate (Corcoran et al., 2009; Zhao et al., 2015), zinc chloride ($ZnCl_2$) (Liebezeit and Dubaish, 2012), zinc bromide (ZnBr) (Quinn et al., 2016), calcium chloride (CaCl) (Stolte et al., 2015), canola oil (Crichton et al., 2017), and lithium metatungstate (Masura et al., 2015).

Imhof et al. (2012) designed the Munich Plastic Sediment Sampler (MPSS), a tool that uses large amounts of $ZnCl_2$ solution to separate particles and is capable of separating out smaller microplastics and samples can be subsampled with replicates (Ling et al., 2017). Nuelle et al. (2014) developed a technique which first used a dense NaCl solution to extract microplastics from the sediments followed by a final NaI flotation step on the reduced sample which has been successfully applied with positive results however, NaI is extremely toxic, which could be a limiting factor for monitoring. Claessens et al. (2013) created an elutriation column to use as a pre-treatment whereby a sediment sample is perturbed by a constant flow of water allowing the separation of the less dense particles.

The aim of the study was to test the efficiency of sampling and extraction methodologies to inform a future project that will examine microplastics in Galway Bay. The comparison of widely applied sampling tools and techniques will assist in the identification of economical advantageous options for future monitoring (Frias et al., 2018). While there are several effective methodologies being applied to both collect and process subtidal sediments for microplastics there have been no comparisons made, that the authors are aware of, between a) sediment sampling tools in regard to microplastic sampling and b) density separation techniques. The authors chose the most widely available and applied tools for the collection of soft sediment i.e. a van Veen day grab, a box corer and a gravity corer. On examination of the current commonly used extraction techniques the authors decided to compare the elutriation column without a subsequent second processing step and the density separation via NaCl both of which provide non-toxic and economically viable options for future monitoring. In addition, a new dense liquid separation method using sodium tungstate dihydrate was also tested and compared to the other extraction methods.

2. Materials and methods

Sampling took place in Galway Bay, whose opening is largely sheltered from the Atlantic Ocean by the Aran Islands, on the west coast of Ireland. The sediments within the bay range from mud to rock (O'Carroll et al., 2017) and the sampling station (53°08′34.8″ 9°27′25.2″W) was chosen due to the soft sediment in that area of the bay (Fig. 1). The sampling was opportunistic and as part of a larger survey being carried out by the *RV Celtic Voyager* in March 2017.

2.1. Sample collection

Three benthic sediment sampling tools, a 3 m gravity corer (Vibro Corer 108 mm) with steel sleeve, box corer (Reineck box corer 20 × 30 cm), and Day grab (van Veen) (0.1 m^2), deployed from the *RV Celtic Voyager*, were used to collect the sediment samples at a depth of 42 m depth. All sediment samples for microplastic analysis were taken from the top 5 cm and stored in glass jars with metal lids. The glass jars had been previously decontaminated by bathing each jar in 10% nitric acid solution and rinsing three times with ultrapure water, as a quality control.

Sediment samples were removed directly from inside each tool after each individual deployment. Each sampling tool was deployed 6 times with the gravity corer being deployed only once. In total, 9 replicates were taken for each tool using a stainless-steel core. The core had a diameter of 5 cm and tape was placed on the outside at 5 cm height to allow for standardisation of the replicates. Similarly, sediment was taken for granulometry from the top 5 cm of the sample, placed in double Ziploc plastic bags, labelled, and stored at −20 °C until processed.

2.2. Particle size analysis

The sediment sample for granulometry and loss on ignition (LOI) was defrosted and transferred into an aluminium tray, homogenized by hand and dried in an oven at 105 °C for 24 h. Granulometry and LOI were carried out according to the protocol of the National Parks and Wildlife Service (NPWS) who manages the Irish State's nature conservation responsibilities (Róisín Nash 2017, pers. comm., 6 Feb.). The protocol is condensed here:

2.2.1. Granulometry

Approximately 35 g of dried sediment was weighed and placed in a glass beaker to which 100 mL of 6% hydrogen peroxide (H_2O_2) solution was added and left to stand overnight in a fume hood. The contents of the beaker were rinsed into a 63 μm sieve. The sample retained on the sieve was washed back into the beaker where sodium hexametaphosphate (SHMP) solution (10 mL, 10%) was added to the beaker and allowed to stand overnight. The mixture was rinsed through a 63 μm sieve with ultrapure water. The retained sample was washed from the sieve into a tray and placed in an oven for drying at 105 °C. When dry, this sediment was sieved through a range of graduated sieves (from