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Review

Recovering microplastics from marine samples: A review of current practices

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ABSTRACT

An important component of microplastic research is development of reproducible methods for microplastic recovery and characterization. Presented is a review of the literature comparing microplastic separation and identification methodologies from seawater, sediment and marine organisms. The efficiency of methods was examined, including processing time, recovery rates, and potential destruction of microplastics. Visual examination and acid digestion were the most common separation methods for seawater samples and organisms, while density flotation was the primary method for sediment. Few studies reported recovery rates, or investigated the physical or chemical impact on plastics. This knowledge gap may lead to misidentification of plastic or unreliable pollution estimates. Further investigation of the impact chemical treatments have on plastic is warranted. Factors, i.e. biomass loading, recovery rates, and chemical compatibility, must be considered to allow for appropriate methodology. Standardizing this will contribute to efficient sample processing, and allow for direct comparison of microplastic contamination across environments.

1. Introduction

Marine plastic pollution has become a global environmental concern and is a growing issue as a result of the exponential increase in the production of plastics. As of 2015, global production of petroleum-based plastics exceeded 300 million metric tons (Avio et al., 2015), with the majority of manufacturing attributed to six main plastic types: polyethylene (PE) (Majewsky et al., 2016), polypropylene (PP) (Majewsky et al., 2016), polyvinyl chloride (PVC), polyurethane (PUR), polystyrene (PS), and polyethylene terephthalate (PET) (Wu et al., 2016). Annual production is estimated to yield a cumulative production of 33 billion metric tons by 2050 (Barrows et al., 2017; Rochman et al., 2013a). One consequence of this mass production is an increased abundance of plastic litter in the ocean and along the shoreline (GESAMP, 2015). It is estimated that 4.8 to 12.7 metric tons of plastic litter enters the ocean environment each year, making this issue one of upmost importance (Andrady, 2011; Barrows et al., 2017). Furthermore, this pollution has the potential to accumulate organic contaminants, such as carcinogenic polychlorinated biphenyls (PCB) (Bellas et al., 2016; Frias et al., 2010; Teuten et al., 2009), polycyclic aromatic hydrocarbons (PAHs) (Rochman et al., 2012; Rochman et al., 2013b) and polybrominated diphenyl ethers (PBDEs) (Tanaka et al., 2012), as well as toxic metals (Nakashima et al., 2011), eventually making its way into and through the marine food web (GESAMP, 2016; Vandermeersch et al., 2015).

Marine plastic pollution has been reported for the past 45 years, and is broadly divided into mega-plastic (> 100 mm diameter), macro-plastic (> 20 mm), meso-plastic (5–20 mm), micro-plastic (< 5 mm) (Barnes et al., 2009; GESAMP, 2016) and nano-plastic (< 100 nm) (Koelmans et al., 2015). Reference to microplastic contamination first appeared in the literature in 1972 (Carpenter et al., 1972), but has only been studied in detail in the past decade or so (Avio et al., 2016; Ivar do Sul and Costa, 2014; Zarfl et al., 2011). The terms 'primary' and 'secondary' microplastics refer to the source, with particles being either specifically manufactured for particular applications (e.g. resin beads, microbeads used in cosmetic products), or produced as a result of fragmentation from larger items (Arthur et al., 2008; GESAMP, 2016). Among the different categories of marine plastic pollution, microplastics are of particular concern due to their ready uptake by marine organisms (Avio et al., 2016; Wright et al., 2013), including some that are consumed by humans, i.e. crabs, oysters, mussels, and fish (Claessens et al., 2013; Cole and Galloway, 2015; Van Cauwenberghe and Janssen, 2014). Indeed, microplastics have been reported from surface waters of every major ocean (Cozar et al., 2014), in sediment types such as intertidal mangroves, beach and deep sea sand (Nor and Obbard, 2014; Quinn et al., 2017; Van Cauwenberghe et al., 2013), and organisms such as bivalves (Li et al., 2016; Vandermeersch et al., 2015) and a wide range of fish species (Guven et al., 2017; Nadal et al., 2016).

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The body of literature investigating the presence and abundance of microplastics in the marine environment has been growing exponentially since the seminal paper by Thompson et al. (2004). However, methods describing the separation and identification of microplastics from environmental samples are highly variable (Hidalgo-Ruz et al., 2012; Shim et al., 2017) preventing robust comparisons of findings across different studies. Existing separation methods include visual separation (Ivar do Sul et al., 2014; Lusher et al., 2014), flotation separation (Frias et al., 2010; Hall et al., 2015), and acid (Claessens et al., 2013; Desforges et al., 2014), alkaline (Tanaka and Takada, 2016; Zhao et al., 2016), oxidative or enzyme digestion (Cole et al., 2014; Courtene-Jones et al., 2017). Many studies, however, do not report on the exact procedures used, nor do they determine the recovery rate of microplastics from digestion methods that have the potential to damage the structure or physical characteristics of plastic polymers (Cole et al., 2014; Quinn et al., 2017). For identification of microplastics, the current recommended method is attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), due to the simplicity of analysis and diagnostic spectral information that it provides (Shim et al., 2017). However, polymer characterization of microplastics using chemical techniques (i.e. FTIR) does not always occur (Baldwin et al., 2016; Ivar do Sul et al., 2014), and is rarely used in the few studies that report on microplastic recovery rates. Importantly, most studies do not report on details such as the time required to process samples, and to separate and identify microplastics from environmental samples, making it difficult to determine the most (cost-)effective and suitable methods for their processing.

In this study, we 1) review the current methods used to separate and identify microplastics in marine environmental samples, i.e. seawater, sediment and marine organisms, 2) describe the sampling and preservation protocols used, 3) provide a synthesis of the separation and identification methods applied and 4) report on the established recovery rates of microplastics, specifically for the commonly reported chemical separation methods that may have an adverse effect on the structural or chemical integrity of plastic items in environmental samples. We also present recommendations to establish reproducible methodologies, including the need for robust testing of chemical separation methods on common plastic pollutants. Implementation of protocols addressing these factors will contribute towards more efficient processing of microplastics from environmental samples, and allow better comparison of microplastic contamination in seawater, sediment and marine organisms.

1.1. Literature search strategy

A systematic literature review was conducted using the search engine Google Scholar and several online databases: Web of Science, PubMed, ScienceDirect and James Cook University's OneSearch (Proquest's Summon 2.0). The iterative search, conducted between December 2016 and April 2017, used various combinations of the following keywords: microplastics, methodology, extraction, isolation, identification, recovery, chemical, enzymatic, digestion, density, flotation, separation, seawater, sediment, biological organisms, and marine pollution. The specific keyword ‘microplastic’ was the primary inclusion criteria. A detailed review of the reference lists of each retrieved article identified additional articles. In total 71 research articles were included within this literature review.

1.2. Seawater samples

Since the first study in 1972 (Carpenter et al., 1972), microplastic particles and fibers have been documented in the surface waters of every major ocean (Cozar et al., 2014). The primary method used for collecting seawater samples is a neuston net tow through the water (Table 1; Supplementary Material Table 1). Originally intended for plankton monitoring, the use of these nets allows for large volumes of water to be sampled with relative ease. Mesh sizes of nets have varied throughout the literature, ranging from 200 μm (Hall et al., 2015) to the most commonly used size of 333 μm, (Brandon et al., 2016; Carpenter and Smith, 1972; Guven et al., 2017; McCormick et al., 2014; Sutton et al., 2016; van der Hal et al., 2017). A mesh size of 333 μm or smaller significantly increases the amount of plastic particles collected (Barrows et al., 2017; Song et al., 2015) but also increases the entrapment of biological biomass. Sampling has been conducted at the surface, subsurface (at an average depth of 3 m) (Cozar et al., 2014), along the benthos (0–2 m above the bottom) (Lima et al., 2014; Morris and Hamilton, 1974) and from ice cores (Lusher et al., 2015).

Apart from neuston nets, a continuous intake system with a mesh filter size ranging from 250 to 300 μm has been used on larger research vessels like those utilized by Enders et al. (2015), Lusher et al. (2014), and Desforges et al. (2014). This method often requires the water sample to travel through multiple mesh filter sizes. For example, Desforges et al. (2014) initially passed samples through a coarse 5 mm filter to remove large debris and organisms, then consecutively through a series of copper sieves of 250 μm, 125 μm and 62.5 μm aperture size. Wastewater management and monitoring relies on different techniques for sampling, including the use of pumps and sieves with a significantly smaller mesh size (12.5 μm); modified versions of this method have been implemented by Majewsky et al. (2016), Dyachenko et al. (2017), and Mintenig et al. (2017) for seawater samples. The potential for loss of microplastics, i.e. trapped in the mesh filters, has yet to be established, with recovery rates for microplastics at each filtration step largely unknown, although filter specifications may provide some insight. Lusher et al. (2014) did, however, demonstrate that by stacking replicate 250 μm mesh sieves followed by a visual assessment, that a single 250 μm mesh sieve was < 100% effective at removing particles from seawater samples. These results suggest an underestimation of microplastic abundance across samples.

The majority of studies do not mention the use of a preservation method (Dyachenko et al., 2017; Gallagher et al., 2016; Majewsky et al., 2016), or specifically state that samples were processed immediately following collection (Cole et al., 2014; Hall et al., 2015; Lusher et al., 2014). The exclusion of a preservation method for seawater samples is acceptable, especially if the primary focus of the study is to recover microplastics, and not the characterization of the biological material (Government du Québec, 2009). However, this has not always been the aim of investigations that sample marine habitats. Historically, reporting of microplastics from seawater samples has been secondary, with sampling and preservation techniques implemented primarily to obtain information on the biological material (Cole et al., 2013; Frias et al., 2014). Preservation techniques are employed to retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source (U. S. Environmental Protection Agency, 1983). This is in direct contrast to the current research into marine pollution, with the primary concern being to quantify microplastics within samples. Nonetheless, some investigations still include biological preservation methods, since maintaining the integrity of the biological matter may still be crucial to other aspects of the study i.e. to establish microplastic:zooplankton ratios (Frias et al., 2014). In these studies, biological preservation methods are generally applied and include using a 4% formalin solution (Frias et al., 2014; Ivar do Sul et al., 2014). If the identification and characterization of the biological material within a sample is not relevant to the study, simple preservation methods such as refrigeration or freezing could be used, if any, since the degradation of the organic material to liberate microplastics is actually preferred.

The critical aspect of microplastic research relates to the separation of microplastics from the biological biomass (i.e. plankton). Flotation separation methods have been widely used for the isolation of microplastics from seawater samples, either standalone (flotation) (Carpenter et al., 1972), with elutriation (Claessens et al., 2013), combined with a hypersaline solution (density flotation) (Hall et al., 2015; Lima et al.,