ARTICLE IN PRESS

MPB-07982; No of Pages 7

Marine Pollution Bulletin xxx (2016) xxx–xxx

Contents lists available at ScienceDirect

Marine Pollution Bulletin

journal homepage: www.elsevier.com/locate/marpolbul

Research paper

A standardized method for sampling and extraction methods for quantifying microplastics in beach sand

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ARTICLE INFO

Article history:
Received 13 July 2016
Received in revised form 19 August 2016
Accepted 22 August 2016
Available online xxxx

Keywords:
Microplastics
Beach sampling
Extraction
Method standardization
Standard operating procedure

ABSTRACT

Microplastics are ubiquitous in the environment, are frequently ingested by organisms, and may potentially cause harm. A range of studies have found significant levels of microplastics in beach sand. However, there is a considerable amount of methodological variability among these studies. Methodological variation currently limits comparisons as there is no standard procedure for sampling or extraction of microplastics. We identify key sampling and extraction procedures across the literature through a detailed review. We find that sampling depth, sampling location, number of repeat extractions, and settling times are the critical parameters of variation. Next, using a case-study we determine whether and to what extent these differences impact study outcomes. By investigating the common practices identified in the literature with the case-study, we provide a standard operating procedure for sampling and extracting microplastics from beach sand.

1. Introduction

Human plastic consumption has increased at a dramatic rate over the last decades. In 1979, global plastic consumption was estimated to be 62 million tonnes, which increased to 160 million tonnes by 2000 (Pardos Marketing, 2006). More recently, global production rose 4% between 2013 and 2014, from 299 million tonnes to 311 million tonnes (Plastics Europe, 2015). A significant proportion of this plastic enters the environment; Jambeck et al. (2015) estimated it to be over 8 million tonnes of plastic enter the marine environment annually. As of 2014, it has been estimated that between 15 and 51 trillion particles, weighing up to 236,000 tons have accumulated in marine systems (van Sebille et al., 2015). One subgroup of emerging concern are small pieces of plastic, termed “microplastics” (Thompson et al., 2004). These are further subdivided into two groups. Primary microplastics are purposefully produced as micron-sized particles, and secondary microplastics are fragments from the breakdown of larger plastic debris. Due to the longevity of plastics, their fragmentation and accumulation in the environment have been considered as one of the most profound and long-lasting recent changes to the Earth's surface (Barnes et al., 2009). The small size of microplastics make them available to both vertebrates and invertebrates. Microplastic ingestion has been observed in a wide variety of organisms, including zooplankton (Cole et al., 2013; Desforges et al., 2015; Setala et al., 2014), filter feeders, such as oysters and mussels (Cole and Galloway, 2015; Van Cauwenberghe et al., 2015a; Van Cauwenberghe and Janssen, 2014), and fish (Lusher et al., 2013; Mazurais et al., 2015).

There is a growing body of literature investigating microplastic pollution on beaches, including industrial plastic beads or granules, plastic fragments, and plastic fibres (Van Cauwenberghe et al., 2015b). While many studies identify the presence of microplastics in the environment, there remain large inconsistencies in their sampling, extraction, and consequent quantification (Shim and Thompson, 2015). Another recent study emphasized the inconsistency in microplastic sampling and extraction techniques, and stressed how current studies are often incomparable as a result (Van Cauwenberghe et al., 2015b). This variation in sampling and extraction processes throughout the scientific literature can potentially prevent comparison across studies (Cole et al., 2011). The variation makes it difficult to perform spatial and temporal distribution analysis, limiting our understanding of the overall microplastic pollution on beaches. This is particularly apparent with studies quantifying microplastics in beach sand, as shown in a recent methodological review (Hidalgo-Ruz et al., 2012). For example, there is considerable variation in the maximum depth during sampling. A number of studies sample the top 1 cm of sand alone (Baztan et al., 2014; Liebezeit and Dubaish, 2012). Others sample the top 5 cm (Heo et al., 2013; McDermid and McMullen, 2004). Due to the current lack of knowledge regarding transportation of microplastics, the different depths used for sampling may determine the abundances recorded. Furthermore, there is an overt problem of varying units throughout the literature (Van Cauwenberghe et al., 2015b). Microplastics have been reported

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http://dx.doi.org/10.1016/j.marpolbul.2016.08.055
0025-326X/

as a quantity per area (Hidalgo-Ruz and Thiel, 2013), per volume (Baztan et al., 2014) and per weight (Dekiff et al., 2014). Although conversions are sometimes possible, the density of the sampled sand may have to be estimated, and if wet weight is used it cannot be compared with dry weight.

Similarly, there are differences in extraction procedures of plastics from beach samples. Although studies generally follow a similar method of extraction by flotation in a dense salt solution (Thompson et al., 2004), a number of parameters within this process vary greatly, or are not defined. Furthermore, a review of 44 studies on beach sand microplastic quantification revealed that the stirring and settling times defined for microplastic extraction varied widely. Similarly, the filtration process, including extracting the supernatant, was frequently unspecified (Hidalgo-Ruz et al., 2012). While some studies mention repeat extractions to improve extraction effectiveness, in most studies this is not included (Browne et al., 2011; Claessens et al., 2011).

In addition to the methodological inconsistencies, there has been a lack of consensus regarding the size range of microplastics. The European Marine Strategy Framework Directive (EMSFD) suggests that the upper bound for microplastic size should be plastic items <5 mm in their largest dimension (Galgani et al., 2013). Although many studies do identify microplastics as plastic material <5 mm, they do not specify dimension sizes, leaving definitional ambiguity (Baztan et al., 2014; Galgani et al., 2013; Heo et al., 2013; Liebezeit and Dubaish, 2012; Martins and Sobral, 2011). Several studies consider 1 mm to be the maximum size, and generally they also omit dimension sizes (Browne et al., 2011; Claessens et al., 2011; Dekiff et al., 2014; Van Cauwenberghe et al., 2013; Vianello et al., 2013).

To overcome the lack of comparability between quantitative studies on microplastics on beaches and to allow cross-examination, our study aims to provide guidance to develop a standardized methodology for microplastic sampling and extraction. This work consists of three parts: firstly, we perform a detailed literature review to identify key differences in beach microplastic sampling and extraction procedures; secondly, in order to determine whether these differences impact study outcomes a case study at Meijendel beach (the Netherlands) is performed; finally, we synthesise our findings and provide a standard operating procedure for future beach microplastic investigations.

2. Materials and methods

2.1. Literature review

A detailed literature review was conducted to identify key methodological procedures in need of standardization in both sampling and extraction of beach sand. Only primary studies assessing microplastic pollution (or equivalent) in beach sand were considered. Studies included were peer-reviewed and published up until 31/12/14. Searches were made with Web of Science [v5.19] using the following keywords: "microplastic pollution", "microplastics beach", "microplastics beaches", "microplastics sediment", "plastic fragments beaches", "plastic debris beaches", and "plastic fragments sediment". A total of 22 studies were identified as meeting the selection criteria (Table 1).

We split the findings into sampling procedures and extraction procedures. Data regarding the variability in sampling procedures included: *microplastic size definition*, *beach zones sampled*, *sample size*, and *sample depth.* Data regarding variability in extraction procedures included: *sample drying temperature/duration*, *settling time*, *number of repeat extractions*, and *quantitative units.* The sampling and extraction procedures were then analysed and compared in terms of methodological variability.

2.2. Case-study

The case-study design was dependent on the findings of this literature review. As such, we list the key findings here, and outline findings further in the results section. In order to determine whether and to what magnitude these literature-identified variations in sampling and extraction procedures influenced study outcomes, a case-study was conducted in Meijendel, the Netherlands (Fig. 1). The predominate direction of the current near the beach is north-eastwards along the coast, while the predominant wind direction is southwest. Sand was collected at the

Table 1
Summary on sampling and extraction methodology used in beach sand sampling.

		Size	Sampling			Extraction				
Location	Study references	definition	Beach zone[a]2b	n	Sampling depth (cm)	Drying duration/Temp (°C)	Extraction process	Stirring time (min)/speed (rpm)	Settling time (min)	Repeat extractions
UK	Thompson et al. (2004)	N/D	ITA	N/D	N/D	N/D/N/D	Flotation	0.5 min / N/D	2	N/D
Hawaii	McDermid and McMullen (2004)	1–15 mm	HTL/SLZ	2	5.5	N/D/N/D	Flotation	1 / manually	N/D	N/D
Singapore	Ng and Obbard (2006)	N/D	HTL	4–8	1, 10–11	N/D/N/D	Flotation	1 / 200	360	3
India	Reddy et al. (2006)	N/D	ITA	10	5	N/D/N/D	Flotation	60–120 / N/D	15	N/D
Brasil	Costa et al. (2010)	≤1 mm	HTL	9	2	Overnight/100	Sieving only	N/A	N/A	N/A
Portugal	Frias et al. (2010)	<5 mm	HTL	N/D	2	N/D/N/D	Flotation	N/D / N/D	N/D	N/D
UK	Browne et al. (2010)	<1 mm	HTL	30	3	N/D/N/D	Flotation	N/D / N/D	N/D	N/D
Belgium	Claessens et al. (2011)	≤1 mm	HTL/ITA	N/D	N/D	N/D/N/D	Flotation	1 / N/D	60	2
Malta	Turner and Holmes (2011)	N/D	Random	11–29	N/D	N/D/N/D	Sieving only	N/A	N/A	N/A
Portugal	Martins and Sobral (2011)	≤5 mm	HTL	6	2	N/D/N/D	Flotation	N/D / N/D	N/D	N/D
Germany	Liebezeit and Dubaish (2012)	<5 mm	N/D	13–15	1	N/D/70	Flotation	N/D / N/D	N/D	3
Chile	Hidalgo-Ruz and Thiel (2013)	<1 mm	HTL	6	2	N/D/N/D	Sieving only	N/A	N/A	N/A
South Korea	Heo et al. (2013)	<5 mm	HTL/CS	10–49	5	N/D/N/D	Sieving only	N/A	N/A	N/A
India	Jayasiri et al. (2013)	<5 mm	HTL	3	2	N/D/N/D	Flotation	N/D / N/D	N/D	N/D
Italy	Vianello et al. (2013)	≤1 mm	N/A	2	0–5	N/D/90	Flotation	1.5 / N/D	60	3
Brasil	Fisner et al. (2013)	N/D	SLZ	10	0–100[b]	N/D/N/D	Flotation	N/D / N/D	N/D	N/D
Canary Islands	Baztan et al. (2014)	<5 mm	HTL	35–88	1	N/D/N/D	On-site flotation	N/D / N/D	N/D	N/D
Norderney	Dekiff et al. (2014)	<5 mm	HTL	12	3	N/D/60	Flotation	N/D / N/D	N/D	N/D
Canada	Mathalon and Hill (2014)	<5 mm	HTL/MTL/LTL	N/D	3–4	N/D/65.5	Flotation	1–2 / N/D	3-6 min.	2
Canada	Castaneda et al. (2014)	<5 mm	N/A	6	10	N/D/N/D	Sieving only	N/A	N/A	N/A
Romania	Popa et al. (2014)	N/D	N/D	3	N/D	N/D/N/D	Flotation	N/D / N/D	N/D	3
Slovenia	Laglbauer et al. (2014)	<5 mm	HTL/ITA	3	5	24 h/100	Flotation	2/manually	30	2

[a] HTL = High Tide Line (including shore line and tidal mark), MTL: Mid Tide Line, LTL = Low Tide Line, ITA = Intertidal area, SL = Shoreline, SLZ: Supralittoral zone, CS: Cross section.
[b] In 10 cm bands.

Please cite this article as: Besley, A., et al., A standardized method for sampling and extraction methods for quantifying microplastics in beach sand, Marine Pollution Bulletin (2016), http://dx.doi.org/10.1016/j.marpolbul.2016.08.055