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Extraction, enumeration and identification methods for monitoring microplastics in the environment

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ABSTRACT

There is much research on the occurrence, pollution characteristics and impacts of microplastics in the marine environment but this omits factors which play important roles in the analysis of microplastics. This review summarizes the methods and techniques in the extraction from sediment, seawater and organisms, and assesses their advantages and limitations according to different experimental conditions, such as salt solution and reagents added to remove organic matter. Similarly, this overview includes the enumeration methods of microplastics by many kinds of microscopes (e.g. stereomicroscope, fluorescent microscope, scanning electron microscope). Advantages and challenges of using micro-FTIR, ART-FTIR, FPA-FTIR, Pry-GC/MS, and Raman spectroscopy in the identification methods are also discussed. This review suggests that monitoring microplastics needs standardized protocols for extraction, identification and quantification and that further research on the effects of microplastics to human health is needed.

Contents

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1. Introduction

Plastic has been acting as one of the fastest-growing parts of the urban waste. Notably, it is estimated that plastic debris accounts for 60–80% of marine debris (Moore, 2008). In recent years, plastic debris has been particularly concerned and identified as an important pollutant in the marine environment. It has accumulated in beaches (Stoiev and Turra, 2016), seawater (Zarfl et al., 2011), even in the deep sea (Van Cauwenberghe et al., 2013b). Plastic debris is divided into microplastics, mesoplastics, and macroplastics (Fendall and Sewell, 2009; Browne et al., 2010). The size range of microplastics is commonly defined as the diameter less than 5 mm, though 1 mm (Browne et al., 2010; Van Cauwenberghe et al., 2013a) or 10 mm (Carson et al., 2011) have also been proposed. Besides, the types of microplastics are various, include polyethylene, thermoplastic polyester, polyvinyl chloride, polypropylene, polystyrene, polycarbonates and nylon.

Studies on the abundance and composition of microplastics have been investigated in Asia (Qiu et al., 2015), Europe (Van Cauwenberghe et al., 2015), Africa (Nel and Froneman, 2015), Oceania (Reisser et al., 2013) and North America (Zbyszewski et al., 2014). Further assessment of the extent of microplastics pollution in one certain region should be carried out by analyzing samples from sediment, seawater and organisms. However, some factors ignored by researchers may play important roles in the analysis of microplastics, for example, there is no standardized protocol for sampling procedures and further analysis. There are many differences, including stirring time, dried temperature, and counting methods, in the processing of sediment samples. All these could cause uncertainty when comparing the abundance of microplastics between different areas. Thus, it is urgent to improve the present analytical methods and create the complete experimental standard and comparable data.

In current research on microplastics, the techniques used in their extraction, enumeration and identification differ. Therefore, this paper describes the extraction, enumeration and identification methods and summarizes relative factors in the experiments, and their advantages and limitations. By standardizing these, we aim to promote the study of monitoring microplastics in the environment.

2. Extraction of microplastics

2.1. Sediment samples

2.1.1. Sampling

Sediment samples are generally taken from beaches, estuaries and the seabed. Sampling on the beaches varies widely from the sampling location, stationing route to sampling depth. First of all, the sampling location could be far away from the center of human activities and lies between coastline and tide line, especially in parallels to the high tide line (Martins and Sobral, 2011; Liebezeit and Dubaish, 2012; Dekiff et al., 2014). The importance of sampling sites is illustrated by Mathalon and Hill (2014), who found that the highest abundance of microplastics was near the high or low tide lines. The stationing route of sampling usually passes along the coastline. Random sampling in a selected area would also be chosen.

There is a wide range of sediment sampling depths, such as 1 cm (Browne et al., 2011; Liebezeit and Dubaish, 2012), 3 cm (Mathalon and Hill, 2014), 5 cm (Van Cauwenberghe et al., 2013a; Corcorana et al., 2015) and 10 cm (Ng and Obbard, 2006). It is expected that different pollution characteristics would be obtained by collecting samples from different depth sediments but there is little research available. Bottom sediment samples could be collected by a box corer (Corcorana et al., 2015), while samples of the surface could be scooped out using iron spoons or non-plastic sampling spades. Then the sediment samples are put into a glass container or aluminum foil.

2.1.2. Pretreatment

After conducting the previous steps, samples must be dried until a constant weight. The drying process does proceed at different temperature, including 50 °C (Qiu et al., 2015), 60 °C (Nor and Obbard, 2014), 70 °C (Liebezeit and Dubaish, 2012), 90 °C (Vianello et al., 2013), even as to −60 °C (Mathalon and Hill, 2014). Samples can also be air dried (Jayasiri et al., 2013). However, microplastics would become deformed and further fragment in the next steps (e.g. elutriation), after heating at high temperature. Based on the heat deflection temperature (HDT) of plastics shown in Table 1, samples oven dried at less than 60 °C can also be a good technique (Osswald et al., 2006).

Then samples are sieved on a mesh (<5 mm) so that the flotation could be handled more easily (Corcorana et al., 2015). H_2O_2 is added to remove organic matter remaining on the surface of microplastics. It has been shown that the ideal solution to dissolve biogenic matter is 35% H_2O_2, compared with solution of 37% HCl and of NaOH with concentrations of 20, 30, 40 and 50% (Nuelle et al., 2014). Moreover, Tagg et al. (2015) found 30% H_2O_2 could also promote the efficiency of filtration and help to identify different microplastics types by FTIR. In addition, Cole et al. (2014) developed an enzymatic digestion protocol that is of great efficacy to reduce the biological materials, comparing with acid and alkaline digestion and this protocol could be carried out at a neutral pH and moderate temperature. In contrast, oxidizing acids (e.g. H_2SO_4, HNO_3) can destroy some type of plastic (e g. polystyrene and nylon) with low pH tolerance. Similarly, alkali could damage and discolored several types of plastics (e.g. nylon, polyethylene), although it can also destroy biological tissue by cleaving proteins, carbohydrates and fats. Although removing organic matters help in visual observation and spectral identification, biofouling is important to assess how long microplastics remain in the environment and sorbed pollutants could be oxidized.

Table 1
Classes of plastics and relative properties.

Plastic class	Material acronyms	Heat deflection temperature (°C)	Density (g/L)
Acrylonitrile butadiene styrene	ABS	95–105	1.06
Acrylonitrile/methyl Methacrylate	AMMA	73	1.17
Acrylonitrile-styrene-acrylate	ASA	95–105	1.07
High-density polyethylene	HDPE	~50	0.96
Low-density polyethylene	LDPE	~35	0.92
Polypropylene	PP	55–70	0.905
Polystyrene	PS	65–85	1.05–1.07
Polyvinyl chlorid	PVC	65–75	1.35–1.39
Polyvinyl butyral	PVB	–	1.1–1.2