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A comparison of microscopic and spectroscopic identification methods for analysis of microplastics in environmental samples

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ABSTRACT

The analysis of microplastics in various environmental samples requires the identification of microplastics from natural materials. The identification technique lacks a standardized protocol. Herein, stereomicroscope and Fourier transform infrared spectroscope (FT-IR) identification methods for microplastics (<1 mm) were compared using the same samples from the sea surface microlayer (SML) and beach sand. Fragmented microplastics were significantly ($p < 0.05$) underestimated and fiber was significantly overestimated using the stereomicroscope both in the SML and beach samples. The total abundance by FT-IR was higher than by microscope both in the SML and beach samples, but they were not significantly ($p > 0.05$) different. Depending on the number of samples and the microplastic size range of interest, the appropriate identification method should be determined; selecting a suitable identification method for microplastics is crucial for evaluating microplastic pollution.

1. Introduction

Microplastics have been recognized as emerging marine pollutants of significant concern, due to their persistence, ubiquity (Kubota, 1994) and toxic potential (Endo et al., 2005; Engler, 2012). Large plastic debris disintegrates and becomes smaller (<1 mm) microplastics, by photolytic, mechanical and biological degradation processes in the marine environment (Browne et al., 2007; Andrady, 2011; Cooper and Corcoran, 2010). Bioavailability increases with the decrease in size of plastic debris (Gregory, 2009), and microplastics have a greater likelihood of absorbing and desorbing toxic chemicals, due to their increased surface area (Lee et al., 2014). Investigation of their negative impacts on marine environments is based on quantification and qualification of microplastics. For this, it is essential to use reliable analytical methods.

Analysis of microplastics from various environmental samples requires a series of procedures including sampling, separation, cleanup and identification. Although several studies on method development and/or comparison for sampling (Norén, 2007, 2011; Song et al., 2014), separation (Imhof et al., 2012; Claessens et al., 2013), cleanup (Claessens et al., 2013) and identification (Vianello et al., 2013), have been carried out, it is still critical to improve methods to yield more precise and accurate results. Among these identification methods, the most widely used should be evaluated for their relevance to future studies. Recently, small-sized microplastics have been found in the marine environment (Thompson et al., 2004; Frias et al., 2010) and the abundance of microplastics increased exponentially with decreasing particle size (Song et al., 2014). The smaller microplastics are more difficult to identify. Ambiguous characteristics of non-plastics (resembling plastics) and plastics (resembling non-plastics) make it difficult to accurately identify microplastics.

The identification of microplastics using three methods has been investigated. First, only the naked eye and/or microscope (McDermid and McMullen, 2004; Costa et al., 2010; Norén, 2007; Collignon et al., 2012; Boerger et al., 2010; Lindborg et al., 2012; Heo et al., 2013) were used to identify microplastics, and some studies included microplastics of <1 mm. Second, microscope (and the naked eye) and instruments were used together (Martins and Sobral, 2011; Doyle et al., 2011; Nor and Obbard, 2014). The microplastics were identified mainly using a microscope or the naked eye and a limited number of selected particles were identified by a spectroscopic method using a Fourier

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transform infrared spectroscope (FT-IR). Third, all of the counted microplastic particles were identified by a spectroscopic method using an FT-IR or a Raman spectroscope (Vianello et al., 2013; Browne et al., 2011; Ng and Obbard, 2006; Song et al., 2014). However, prior to this study there has been no comparison of the advantages, disadvantages or accuracy of methods of microplastics identification.

In this study, we aimed to assess the microscopic and spectroscopic identification of microplastics. Specifically, we addressed the following questions: (1) How different are the abundances of microplastics determined using the microscopic and spectroscopic identification methods? (2) If the abundances of microplastics differ between the two methods, which component contributes to the measurement error? For the comparison, stereomicroscope and FT-IR microscope identification methods were applied to the same samples from surface microlayer water and beach sand. In addition, microplastic abundances were compared according to type (fragment, fiber, sheet and expanded polystyrene (EPS)) and size.

2. Materials & methods

2.1. Microplastic sampling

2.1.1. Surface microlayer

The microplastics sampling region and methods from the sea surface microlayer (SML) have been published elsewhere (Song et al., 2014) in detail. Water samples were collected near- and offshore of Geoje Island, which receives riverine discharge from the nearby Nakdong River, South Korea (Fig. 1). The SML water samples were collected at 10 stations in May and July, 2012 (Song et al., 2014). A metal sieve was used for SML water samplers, which typically collected SML at a depth range of 150–400 μm (Cunliffe et al., 2013). The microplastics and the SML water were trapped within the metal sieve mesh spaces by surface tension. A 2-mm mesh sieve of 20-cm diameter was placed in contact with the sea surface 100 times, covering a 3.14-m^2 sampling area at each station. The water trapped within the mesh spaces was collected in the stainless tray and transferred to a 1-L polyethylene bottle. The final volume of SML water sampled per station was in the range of 2.2–2.8 L.

2.1.2. Sand beach

Sediment samples were collected from six beaches on Geoje Island, which are affected by riverine discharge from the nearby Nakdong River, in May, 2012 (Fig. 1). At each beach, 10 positions were randomly selected along the high strandline. About 12.5 L of sand samples were collected in a 0.5×0.5 m quadrat with 5-cm depth, using a stainless scoop, through a 1-mm sieve (Tyler sieve, CISA, Spain). The sieved sand samples were well mixed in a stainless tray and 1 L of sand was transferred to a polyethylene bottle. In the case of wet sand samples, sieving was conducted after air-drying in the laboratory to avoid contamination of the mesh screen cover.

2.2. Analysis of microplastics

2.2.1. Microscopic analysis

The SML samples were filtered (GF/F; 0.75 μm; 47 mm ∅) in the laboratory. The filter papers were dried at 60 °C and stored in Petri dishes. The microplastics on the filter paper were identified and counted using a stereomicroscope (ZEISS Model Discovery "SV8"). Microplastics were categorized into four types (fragment, fiber, sheet and expanded polystyrene (EPS)) and again according to six maximal length classes (<50, 50–100, 100–200, 200–500,

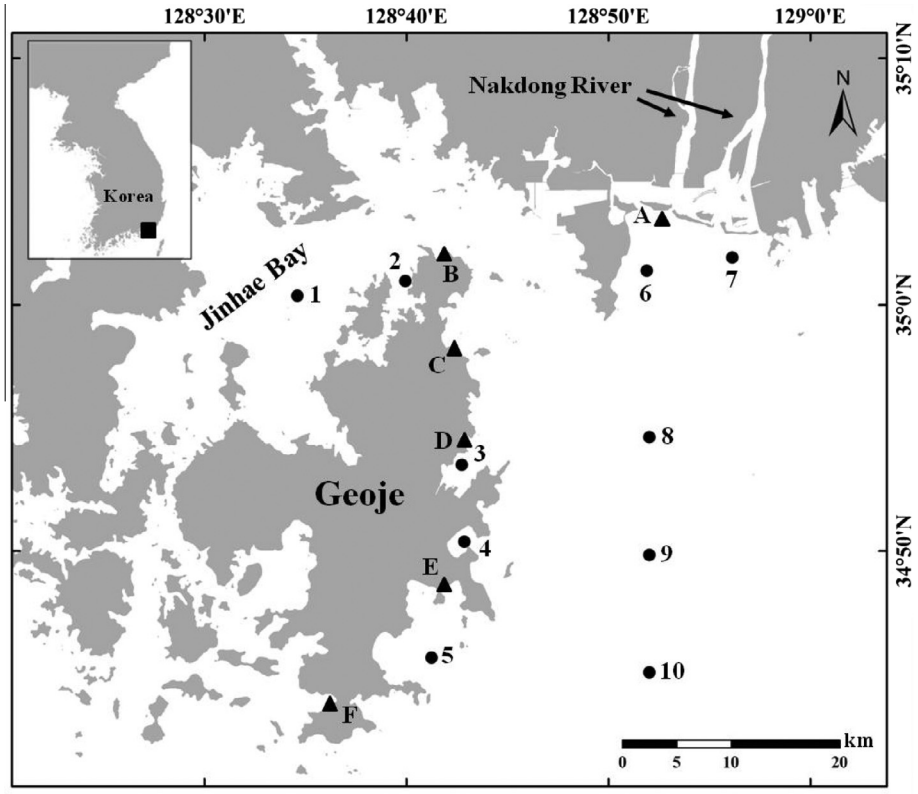

Fig. 1. Map showing the locations of microplastic sampling stations for surface microlayer water (● – Stations 1–10) and beach sediment (▲ – Stations A–F: A; Jinwoodo, B; Guyoung, C; Heungnam, D; Deokpo, E; Wahyun and F; Myoungsa) around Geoje Island, South Korea.

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