



Formation of microscopic particles during the degradation of different polymers



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HIGHLIGHTS

- Degradation of seven polymers in aquatic medium results in an increased formation of microscopic plastic particles.
- Weathering of polystyrene and polylactic acid generated the highest number of particles, especially in the nanometer range.
- Surface erosion of all seven materials produced particles from 30 nm up to 60 μm .
- Particle concentration increased with decreasing particle diameter.
- Degradation processes start with chain scission at the polymer surface releasing many very small particles.

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ABSTRACT

This study investigated the formation and size distribution of microscopic plastic particles during the degradation of different plastic materials. Particle number concentrations in the size range 30 nm–60 μm were measured by nanoparticle tracking analysis (NTA) and Coulter Counter techniques. Each of the plastics used exhibited a measurable increase in the release of particles into the surrounding solution, with polystyrene (PS) and polylactic acid (PLA) generating the highest particle concentrations. After 112 d, particle concentrations ranged from 2147 particles ml^{-1} in the control (C) to 92,465 particles ml^{-1} for PS in the 2–60 μm size class; 1.2×10^5 particles ml^{-1} (C) to 11.6×10^6 for PLA in the 0.6–18 μm size class; and 0.2×10^8 particles ml^{-1} (C) to 6.4×10^8 particles ml^{-1} for PS in the 30–2000 nm size class (84 d). A classification of samples based on principal component analysis showed a separation between the different plastic types, with PLA clustering individually in each of the three size classes. In addition, particle size distribution models were used to examine more closely the size distribution data generated by NTA. Overall, the results indicate that at the beginning of plastic weathering processes chain scission at the polymer surface causes many very small particles to be released into the surrounding solution and those concentrations may vary between plastic types.

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1. Introduction

It has recently been shown that indicators of anthropogenic activity in lake sediments include a complex combination of materials, including plastics, and that these indicators differ greatly from Holocene signatures (Waters et al., 2016). As such, the environmental degradation of different plastic materials is of great interest. Conventional understanding regarding plastic degradation processes is derived from laboratory studies that often investigate a

single mechanism of degradation such as photo-, thermal-, or bio-degradation (Lambert et al., 2013b). These studies have tended to focus on thin films and characterise degradation in terms of weight loss, changes in tensile strength, breakdown of molecular structure, and the identification of specific microbial strains to utilize specific polymer types. Presently, little is known about the formation of microscopic particles during the degradation of different plastic materials. This is an important issue given the current interest in the environmental occurrence and effects of microplastics that are generally classified as having a diameter less than 5 mm (GESAMP, 2015). In addition, the formation of particles below the threshold of visible detection, such as those at the nanoscale, during plastic degradation is also considered highly likely (Andrady, 2011; Lambert et al., 2014; Mattsson et al., 2015).

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Analytical methods for particle characterisation include Coulter Counter and nanoparticle tracking analysis (NTA). Coulter Counter is a conductivity based technique that is able to measure particles ranging from 0.4 to 1200 μm diameter depending on the size of aperture used (Rhyner, 2011). The sample of interest is diluted in a weak electrolyte solution and is then passed through the aperture concurrent with an electrical current. The voltage applied across the aperture is known as the sensing area and as particles pass through the aperture they displace their own volume of electrolyte and alter the impedance (Demeule et al., 2010; Narhi et al., 2009). This change in impedance produces a pulse that is directly proportional to the volume of the particle and this converted signal enables the instrument to calculate particle size and size distribution (Demeule et al., 2010; Narhi et al., 2009).

NTA visualizes, measures, and characterises particles in the 30–2000 nm size range through the use of a laser beam to illuminate particles (Filipe et al., 2010). The NTA technique is based on the tracking of single particles and the software relates the rate of Brownian motion to particle size. The scattered light is captured using a digital camera and the motion of each particle is tracked from frame to frame by the NTA software (Kramberger et al., 2012). The rate of particle movement is related to a sphere equivalent hydrodynamic radius as calculated through the Stokes-Einstein equation and particle size is observed on a particle-by-particle basis (La Rocca et al., 2014). The principles of NTA are further described in Hole et al. (2013), Gillespie et al. (2011), and Gallego-Urrea et al. (2010). The NTA platform has previously been used to investigate the particle formation during the degradation of natural rubber latex (Lambert et al., 2013a) and polystyrene (Lambert and Wagner, 2016).

The size distribution of particles in aquatic environments is an important property that influences many natural processes, such as the scattering and absorption of light in the water column, the exchange of substances between solid and liquid phases, and the transportation of substances to sediments, as well as their interactions with biological processes (Jonasz, 1983; Reynolds et al., 2010). A particle size distribution (PSD) can be defined as the average number of particles within a given size classification (Reynolds et al., 2010). In addition, modelling the size distribution of particles is often useful when highly variable particle populations are expected as they can provide a more complete description of PSD properties (Hwang et al., 2002). Therefore, an investigation of PSD of plastic particles formed during degradation processes is of particular interest, and representing the particle concentration data as a mathematical function may provide an opportunity to search for materials with similar degradation profiles.

In this study, we aimed to investigate the formation of microscopic particles during the degradation of different plastic materials, under aqueous conditions. To do this, a weathering chamber was used to accelerate the degradation process, and seven plastic materials were compared that represented five different polymer types. The materials selected consisted of two plastic pellets (polyethylene (PE) and polypropylene (PP)), and five consumer plastics including two types of PP packaging materials, a polystyrene (PS) coffee-to-go lid, a polyethylene terephthalate (PET) water bottle, and a polylactic acid (PLA) beverage cup. The main objective was to characterise the formation of particles from 30 nm to 60 μm in diameter and to explore the effects of polymer type on particle formation. A second objective was to further examine the particle distribution data generated by NTA within the context of commonly used PSD modelling approaches.

2. Methods

2.1. Test materials and their preparation

The plastic pellets used in this work were purchased from a commercial source and the consumer plastics were sourced from commercial retailers in Darmstadt, Germany (supporting information (SI) Table S1). The polymer type of the consumer plastics were identified through their recycling code, and the identification of all materials was further confirmed using ATR-FTIR spectroscopy (Attenuated Total Reflection-Fourier Transform Infra-Red, PerkinElmer UATR Two; the individual spectra are available in the SI Fig. S1). The plastics used were durable materials and not those associated with thin film materials.

The consumer plastics were emptied and plastic material covered by labelling was cut away and not used to exclude any glue residues. The remaining plastic material was then gently washed under a running demineralised water tap, with care taken to prevent artificial disturbance of the material surfaces. After drying the consumer plastics were then cut into 1×1 cm squares using stainless steel scissors. The plastic materials were then placed in glass vials, immersed in 20 ml demineralised water, and placed in a weathering chamber (Binder GmbH). The exposure conditions were kept static with the temperature set to 30 °C and continuous exposure to light in both the visible and ultra-violet (UV; 320–400 nm) range. Samples were taken after 0, 7, 14, 28, 56, 84, and 112 days, for each time point individual samples were established in triplicate alongside control samples (i.e. water only). Evaporation was dealt with by regularly replacing lost water. For the consumer plastics each individual replicate contained one, 1×1 cm square of material, and for the plastic pellets each individual replicate contained five pellets. The average surface area was as follows; 1.96 cm² for the five pellets (0.4 cm² per pellet), 2.03 cm² for the PP film, 2.12 cm² for the PP sheet, and 2.06 cm² for the three remaining materials. A laboratory blank of 20 ml demineralised water was prepared in a fresh glass vessel on the day of each sample collection to document any contamination that may result from the analytical process.

2.2. Analytical methods

To characterise the formation of plastic particles during the degradation of different plastic materials two methods were utilised: (i) Coulter Counter was used to determine the particle concentrations in the 0.6–60 μm size range, and (ii) NTA was used to determine particle size concentrations in the size range of 30–2000 nm. Coulter Counter measurements were performed using a Multisizer 3 equipped with a 30 μm and 100 μm aperture (Beckman Coulter, Fullerton, CA, USA). The 100 μm aperture allows particles with a diameter of 2–60 μm to be counted, and the 30 μm aperture allows for the counting of 0.6–18 μm particles. Liquid samples were diluted between 20 and 50 fold with Isoton II electrolyte (ISO; 0.9% NaCl electrolyte from Beckman Coulter) solution to provide adequate ionic strength for the analysis. To adequately characterise particle concentration each individual sample, control, and blank were analysed in triplicate with each aperture.

NTA analysis was performed using NanoSight LM 10 (NanoSight Ltd, Wiltshire, UK). Only samples generated from days 0–84 were analysed by NTA because of unexpected instrument down time. To characterise each individual replicate sample, control, and blank in a representative manner three video images of each were taken and designated as a single measurement. Video image length was set at 30 s and imaging was performed at room temperature. The video images were then processed using NTA 2.3 software with the following parameters; detention threshold set to auto; blur set to

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