



Pigments and plastic in limnetic ecosystems: A qualitative and quantitative study on microparticles of different size classes



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ABSTRACT

Recently, macroplastic (>5 mm) and especially microplastic (<5 mm) particles have been reported as emerging contaminants in marine and limnetic ecosystems. Their coloration is gained by the addition of pigments to the polymer blend which is the major component of the respective product. However, color is also a feature of paint and coatings whereby the pigment is the major component. Once abraded from a surface, paint particles may enter the environment via similar pathways as microplastic particles.

So far no detailed studies of microplastic particles (pigmented and non-pigmented) as well as paint particles have been performed focusing on very small microparticles (1–50 μm), in either marine or limnetic ecosystems. Using Raman microspectroscopy with a spatial resolution down to 1 μm, we report a remarkable increase in the occurrence of (pigmented) microplastic particles below 500 μm. Among those, most particles were found at a size of ~130 μm in a freshwater ecosystem (subalpine Lake Garda, Italy). Moreover, our qualitative and quantitative analyses revealed that the number of paint microparticles significantly increased below the size range of 50 μm due to their brittleness (the smallest detected paint particle had a size of 4 μm). Inductively coupled plasma mass spectrometry measurements showed that both colored particles found in nature as well as virgin particles contain a high variety of metals such as cadmium, lead and copper. These additives may elicit adverse effects in biota ingesting these microparticles, thus paints and associated compounds may act as formerly overlooked contaminants in freshwater ecosystems.

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1. Introduction

Technical advances are often accompanied by the development of modern and highly innovative materials, chemicals and compounds. One recent change in material use is reflected in plastic production which has grown up to 311 million metric tons per year worldwide (PlasticsEurope, 2015), and has led to a change in the composition of our household and industrial waste. As a consequence, an increase of plastic litter has been reported in marine

ecosystems (Thompson et al., 2009). Plastic waste is introduced into the sea as larger waste fragments (macroplastic particles, >5 mm), is then degraded by UV-radiation and subsequently fragmented through mechanical forces and biological processes into tiny “secondary” microplastic particles (<5 mm, Browne et al., 2007). In contrast pre-production pellets lost during transport or spilled at the production plants belong to “primary” sources (Hidalgo-Ruz et al., 2012). Small microplastic fibers released from synthetic clothing through mechanical processes in washing machines, in addition to “primary” microplastic particles, often used as abrasives in cosmetics or cleaning products, are introduced into the aquatic environment via sewage effluents (Browne et al., 2011; Fendall and Sewell, 2009; McCormick et al., 2014). As a consequence, not only marine habitats are affected. Just recently scientists discovered the existence of microplastic particles in limnetic ecosystems (reviewed in: Dris et al., 2015).

To achieve one appealing feature of plastic products – their

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coloration – the industry adds pigments to the polymer blends whereby the polymer usually remains the major component of the respective product (Freitag and Stoye, 2008). Though colorfulness is not exclusively a feature of plastic products. The utilization of pigments in colorful paintings and coatings is ubiquitous not only for wooden as well as metal items, but also lightweight products made of plastic, fiberglass or carbon fibers (Freitag and Stoye, 2008; Turner, 2010). In this case, the pigment is the major component and the polymer serves as a binder, such as an alkyd for industrial paints (e.g. in marine applications, Shtykova et al., 2006) or a (poly)acrylate/styrene (Song et al., 2014).

In the same manner as plastic particles, pigment coated surfaces are both mechanically weathered and abraded leading to the formation of so called paint particles. Heavy abrasion occurs while sandblasting buildings or in ship maintenance and repair facilities, in both cases generating dust as well as large fragments (Takahashi et al., 2012). Several studies have already reported on paint particles in terrestrial environments (Bogden and Louria, 1975; Sturges and Harrison, 1985). These paint particles can subsequently enter the aquatic ecosystem either directly or indirectly through wind and water run-off, likely with additional fragmentation occurring. Paint particles observed on rooftops in coastal locations were found to travel long distances via wind and water (Turner, 2010). These particles have also been discovered in the marine environment, including tidal inlet sediments along the English coast, the seabed next to marinas in Malta or on the sea surface (Norén and Naustvoll, 2011; Song et al., 2014; Takahashi et al., 2012; Turner, 2010).

Similar to the ubiquitous macro- and microplastic particles, microscale paint particles are prone to consumption by aquatic organisms from different trophic levels and feeding strategies. Ingestion may incur negative effects on the biota, for example through toxic or potentially harmful additives, mixed directly into the polymer blend (Cole et al., 2013; Eerkes-Medrano et al., 2015; Lithner et al., 2011; Wright et al., 2013). Another threat from particle ingestion arises from their ability to adsorb hazardous substances such as persisting organic pollutants (POPs) or toxic metals (Bakir et al., 2012; Holmes et al., 2012). Colored plastic particles containing pigments may supply an additional high burden of metals. Furthermore, other substances associated with paints and coatings may have adverse effects on organisms or the environment. These mixture of hazardous substances, either adsorbed or intentionally added, can be eventually released in the digestive tract of the organisms that swallowed the respective particle(s) (Browne et al., 2013). In addition, very small particles (1 μm –50 μm) pose a higher risk of being incorporated into the organism's tissue (Van Cauwenberghe et al., 2015). Despite the high potential ecological risk of tiny particles, few studies have been able to identify particles with sizes below 500 μm , even fewer yet below 50 μm , from environmental samples (reviewed in: Dris et al., 2015; Hidalgo-Ruz et al., 2012; Ivar do Sul and Costa, 2014; Rocha-Santos and Duarte, 2015; Van Cauwenberghe et al., 2015). The lack of an extensive qualitative and quantitative analysis of microparticles in the environment may arise from the limited number of reliable identification methods. Until now only two methods (micro-FIIR and Raman microspectroscopy, RM) can provide the identification of environmental microparticles with a spatial resolution down to ~20 μm and 1 μm , respectively (Enders et al., 2015; Imhof et al., 2013; Löder and Gerdt, 2015; Van Cauwenberghe et al., 2013).

In this study we confirm for the first time the existence of paint particles in a freshwater ecosystem based on samples from beach sediments of Lake Garda. To get further insights in the size distribution of plastic particles compared to paint particles from freshwater beach sediments we quantitatively and qualitatively assess the composition of microparticles including those below 50 μm by means of Raman microspectroscopy. As metals can adsorb to plastic

particles or can be an inherent load, in a second step additional inductively coupled plasma mass spectrometry (ICP-MS) measurements reveal the elemental composition of sampled plastic particles (pigmented and non-pigmented) and provide a first time assessment of freshwater plastic particle metal contamination. A comparison with the metal burden of unused colored plastic particles may indicate possible sources.

In detail we aimed to analyze: (i) the concentration of non-pigmented and pigmented plastic particles in comparison to paint particles (ii) the size distribution of the particles (especially in the size range of very small microparticles (1–50 μm), (iii) the spatial distribution of plastic and paint particles and (iv) the elemental composition of a subsample of particles compared to unused plastic products.

2. Materials & methods

2.1. Sediment collection

Beach sediment sampling was performed at Lake Garda, Italy. The lake is located at the eastern border of the subalpine region and its water body belongs to one of the key elements of the tourist economy (Salmaso and Mosello, 2010). Moreover Lake Garda is the largest freshwater supply in Italy. The water is used for domestic consumption, agricultural and industrial purposes such as the generation of hydroelectric power and it supports a small but productive fishery (Magni et al., 2008). We collected sediment samples at 5 different beaches along the coastline of Lake Garda, Italy. As the aim of this campaign was the establishment of a standardized sampling method we performed 3 replicates at each sample area within the littoral zone (at the waterline, in the flotsam and at the high water line). Sampling itself was performed using a 20 m transect in combination with 10 sediment cores (diameter 10 cm) to a depth of 5 cm with an interval of 2 m, resulting in a sample volume of approximately 4–6 L of each location. Each sample was stored in a wide neck polyethylene container of 6 L volume. Contamination through abrasion from the container was excluded, since no particles with the identical spectral pattern were detected by RM.

2.2. Particle extraction from Lake Garda beach sediments

The extraction of the particles was performed using the Munich Plastic Sediment Separator (MPSS, Imhof et al., 2012). As separation fluid a zinc chloride solution (ZnCl_2 technical grade, Th. Geyer, Germany) with a density of 1.6–1.7 kg/L was used. This technique enables us to extract all particles with a density lower than 1.6 kg/L with a recovery rate of 100% for macro- and large microplastic particles (>1 mm) and $95.5 \pm 1.8\%$ for small microparticles (<1 mm, Imhof et al., 2012). After introduction of the sample into the MPSS, large fragments (>1 cm) were singled out and thoroughly rinsed with zinc chloride solution in order to return potentially attached particles back into the MPSS. The sediment was stirred for 3 h, then the sediment had 12 h (overnight) to settle down. Similarly the less dense particles had enough time to rise to the surface. Depending on the amount of organic matter e.g. biofilm, algae, leaves, etc., the sample was subsequently (i) directly filtered on a quartz fiber filter (retention efficiency 98% at 2.2 μm , smaller particles will be retained with lower probabilities, Whatman QM-A, GE Healthcare Europe GmbH) and rinsed with hydrogen peroxide and distilled water; (ii) the samples were sieved using a 750 μm mesh into 250 ml centrifuge tubes (Eppendorf AG, Germany). Particles >750 μm were picked with tweezers and stored in petridishes until they were identified using RM. Centrifugation of the fraction with particles <750 μm with slow speed (~1500 rpm) facilitated the

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