Short communication

Microplastics in coastal sediments from Southern Portuguese shelf waters

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A B S T R A C T

Microplastics are well-documented pollutants in the marine environment that result from fragmentation of larger plastic items. Due to their long chemical chains, they can remain in the environment for long periods of time. It is estimated that the vast majority (80%) of marine litter derives from land sources and that 70% will sink and remain at the bottom of the ocean. Microplastics that result from fragmentation of larger pieces of plastic are common to be found in beaches and in the water surface. The most common microplastics are pellets, fragments and fibres.

This work provides original data of the presence of microplastics in coastal sediments from Southern Portuguese shelf waters, reporting on microplastic concentration and polymer types.

Microplastic particles were found in nearly 56% of sediment samples, accounting a total of 31 particles in 27 samples. The vast majority were microfibers (25), identified as rayon fibres, and fragments (6) identified as polypropylene, through infrared spectroscopy (μ-FTIR). The concentration and polymer type data is consistent with other relevant studies and reports worldwide.

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1. Introduction

The global environmental problem that marine litter represents was first documented in the 1970’s (Carpenter and Smith, 1972; Colton et al., 1974), yet it was only in the 2000’s that the topic raised international awareness (Moore et al., 2001). Approximately 80% of marine litter originates from land sources while approximately 20% derives from the sea. Regarding distribution, it is believed that 15% of marine litter is accumulated on beaches and coastal areas, another 15% is floating on the oceans and 70% sinks and remains in sediment ate the bottom of the ocean (UNEP, 2005).

Plastic is the most abundant marine litter category, and due to its characteristics can remain in the environment for long periods of time, eventually causing impacts on wildlife, tourism, fishing and shipping activities (Arthur et al; 2009; Andrady, 2011; Frias et al., 2014; Ivar do Sul and Costa, 2014; Jang et al., 2014; Obbard et al., 2014; Thompson et al., 2004).

A recent study identified the widespread distribution of marine litter in the ocean, with an estimate of at least 5.25 trillion plastic particles currently floating at the sea (Eriksen et al., 2014). This estimate focus mainly on floating plastics and does not take into account materials that sink and accumulate at the bottom of the ocean. There are also studies that show accumulation of plastics and fibres in the deep sea (Cauwenbergh et al., 2013; Schlining et al., 2013; Pham et al., 2014; Vieira et al., 2014; Woodall et al., 2014), at a depth range from 60 to 5000 m. Although studies such as these are relevant, there is still a lack of information about marine litter accumulation in low depth coastal sediments and shallow waters.

Data on marine litter abundance and concentrations in the marine environment is urgently needed to address the European Directive 2008/56/EC (Marine Strategy Framework Directive, heneinafter MSFD). This legal framework is a key element in Europe’s actions to address this issue and calls for all the EU’s marine regions and sub-regions to reach ‘Good Environmental Status’ (GES) by 2020. Marine litter is addressed for the first time in the MSFD in an integrated way towards the protection of the marine environment (Galgani et al., 2013, 2014). Descriptor 10 of the MSFD will establish baseline quantities, properties and potential
impacts of micro-particles, microplastics being the most significant part of this (Galgani et al., 2014; Gago et al., 2014).

The hypothesis and line of thought for this work was related to the presence or absence of microplastic particles in subtidal coastal sediments from Southern Portuguese waters.

The main goals of this study are to (1) quantify microplastic abundance in coastal sediments collected in Southern Portugal (Algarve region) and (2) identify the nature of microplastics using Fourier Transformed Infrared Spectroscopy (μ-FTIR).

2. Materials and methods

2.1. Sample collection and processing

Coastal sediment samples were collected by divers of the Portuguese Task Group for the Extension of the Continental Shelf (EMEPC), during June and August, 2013 as part of the M@rbis campaign, in Algarve coastal waters. In each sampling stations (Fig. 1), a transect of approximately 100 m length and 4 m width was used to collect subtidal coastal sediments (Table 1). A total of 27 samples were collected from 10 transects, including replicates. The vast majority of the coastal sediments were silts and clays.

Samples were preserved in 96% ethanol and stored in closed containers, properly identified. While processing the samples, approximately 1 g of sediment, in triplicate, was retrieved from each sample and introduced in a drying oven for 48 h at 60 °C to determine water content. Samples were introduced into 2 L beakers containing a high density sodium chloride (NaCl; 140 g L⁻¹) solution and stirred vigorously for 5 min, before collecting water to filter. After mixing the sediment will settle to the bottom while microplastics will remain in suspension or floating plastic particles. Recovered microplastics were then examined under a stereoscopic microscope to sort and measure the microscopic plastic particles. Precautions were taken while processing microplastic samples as fibre contamination is likely to occur, particularly having control petri dishes to recover particles from air. Therefore, laboratory blanks were used to exclude fibres that could result from cross contamination.

Particles were photographed and singled out, using tweezers, onto concave slides covered and stored until further analysis. All sample processing (extraction, picking and FTIR analysis) was conducted with extreme care to avoid contamination. Checks for contamination during processing were made by exposing filter paper to the air in the laboratory, whenever samples were open to the laboratory environment. As an additional precaution, those handling the samples wore only natural fibre clothing and were protected with 100% cotton laboratory coats.

2.2. μ-FTIR analysis

The μ-FTIR analysis was performed as described in Frias et al., 2014. Succinctly: micro samples were carefully cut under the Leica KL 1500 LCD microscope, equipped with a 12 × objective and a Leica® Degilux 1 digital camera, with external illumination by optical fibres. Samples were compressed in a diamond anvil compression cell, and infrared spectra were acquired in a Nicolet® Nexus spectrophotometer coupled to a Continuum microscope (32 × objective) with a MCT detector. Spectra were collected in transmission mode in 128 scans, with a resolution of 4 cm⁻¹.

The spectra are shown as acquired, without corrections or any further manipulations, except for the occasional removal of the CO₂ absorption at ca. 2200-2400 cm⁻¹. The identification of polymers was first made by searching the extensive polymer spectral database of the Department of Conservation and Restoration (DCR), and afterwards by comparison analysis of characteristic bands with spectral assignments, in accordance with the literature (Hummel, 2002). The spectra are shown as acquired, without corrections or any further manipulations, except for the occasional removal of the CO₂ absorption at ca. 2200-2400 cm⁻¹. The identification of polymers was first made by searching the extensive polymer spectral database of the DCR, and afterwards by comparison analysis of characteristic bands with spectral assignments.

2.3. Statistical analysis

Data was analysed by non-parametric statistics after invalidation of homogeneity of variances, determined by Levene’s test. The Mann—Whitney U test was used for pairwise comparisons between depth and density of microplastics. The significance level for all analysis was set at 95% (α = 0.05). All calculations were performed using the software Statistica® 7.0 (Statsoft Inc., Tulsa, OK, USA).
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3. Results

3.1. Microplastics in subtidal coastal sediments

Microplastics from coastal sediments of the Algarve region were retrieved from sediment samples, in order to estimate concentrations of microplastics and to identify polymer type. A total of 31 microplastics (25 fibres and 6 fragments) were collected (Table 1; Fig. 2). Fibres collected occurred in 4 different colours (red, green, blue and black) and fragments in two colours (blue and green).

The overall percentage of microplastics per volume of sediment is 0.24%, and the density of microplastics is $0.01 \pm 0.001$ microplastics g$^{-1}$. Most microfibers were collected at a depth beneath 20 m (Fig. 3).

No significant statistical correlation between depth and site of collection or between depth and number of microplastics (either per volume or per weight of sediment) was found possible due to the reduced number of samples.

3.2. μ-FTIR analysis

The vast majority of the samples (25 out of 31) were identified as a semisynthetic cellulose based polymer, commonly known as Rayon (Fig. 4), with a percentage match ranging from 60 to 87%. Identification was possible due to the detection of the infrared characteristic bands of cellulose (Fig. 5): 3650-3000 cm$^{-1}$ (OH stretching modes), 2900-2820 cm$^{-1}$ (CH stretching modes), 1500-1200 cm$^{-1}$ (CH and COH bending modes) and 1200-1000 cm$^{-1}$ (C–O–C stretching and ring vibrational modes).

The remaining 6 microplastic fragments, to which sample ALST 107 R2 (Fig. 6) is an example, were identified as polypropylene with a match of 85–95%, due to the presence of bands at 2960-2835 cm$^{-1}$ (CH$_2$ stretching modes), 1460 cm$^{-1}$ (CH$_3$ asymmetric bending mode) and 1377 cm$^{-1}$ (symmetric bending mode).

4. Discussion

Recent studies on marine litter alert to the rising number of microplastic particles in aquatic environments, while advertizing to the potential toxic effects of these particles on the environment. There are recent evidences that microplastic particles, such as fibres and particles resulting from exfoliants reach the marine environment (Browne et al., 2011; Cole et al., 2011), with unknown consequences for wildlife.

Most available data on microplastics entrapped in sediments is based on studies conducted in intertidal zones and sandy shorelines. A recent review by Hidalgo-Ruz et al. (2012) identified over 40 studies where abundance of microplastics in sandy beaches was assessed. The studies covered different beaches and extensions, from intertidal to supralittoral zones. The majority of studies, however, focused on the most recent marine litter deposited at the high tide line.

This work focus on microplastics from underwater sediment samples from shelf waters, which compared to sampling sediments on beaches, have higher methodological limitations and associated financial constraints. Nonetheless, the separation methodology is similar. Separation of microplastics (fragments and fibres < 500 µm) from bulk sediment by density and filtration methods is more efficient in relatively coarse sediments than in fine materials (silt and clay), as the filters do not clog as fast with coarse sediments. Several separation devices and methodologies have been recently developed to tackle some of the methodological limitations on the laboratories and, there are merits to all these approaches (e.g. the Munich Plastic Sediment Separator, Imhof et al., 2012). However detailed cross calibration of extraction, efficiency, sampling time, equipment costs and health and safety procedures are yet to be undertaken among most methods (Galgani et al., 2013). Despite the recent advances, there are no standard procedures presently available regarding the quantification of microplastics in sediments or water (Andrady, 2011; Galgani et al., 2013).

In this study, a total of 31 microplastic particles were collected from 27 samples, where the vast majority (25) were identified as fibres, with the chemical spectra assignment of Rayon, a semisynthetic cellulose based polymer. Rayon is the oldest commercial manmade fibre composed of regenerated cellulose and its fibres.
possess a range of well-known properties, from flame retardants to super absorbent ability, meeting the demands for a wide variety of uses. Essentially used in personal hygiene products (e.g. wipes) and clothing, rayon fibres may be introduced in the marine environment through wastewater, and one of the main inputs is presumed to be washing machines (Browne et al., 2011; Woodall et al., 2014). Rayon used to be a common fibre in the nonwovens sector, but was gradually replaced by polyethylene terephthalate (PET), and by polypropylene (PP) (Kauffman, 1993; Park et al., 2004).

This predominance of Rayon in sediments is in accordance with recent results published by Woodall et al. (2014) regarding deep sea sediments. Similar proportion were reported in fish (57.8% of synthetic particles ingested) (Lusher et al., 2013) and ice cores (54%) (Obbard et al., 2004). Fibres are often the dominant microplastics found in sediments (Claessens et al., 2011).

Experiments sampling wastewater from domestic washing machines demonstrated that a single garment can produce >1900 fibres per wash (Browne et al., 2011). This suggests that a large proportion of microplastic fibres found in the marine environment may be derived from wastewater as a consequence of laundry activities. While handling and processing samples in laboratory facilities, precautions must be taken into consideration to avoid fibre contamination.

Fragments (6) were identified as polypropylene (PP), one of the most common plastic polymers in worldwide production. In what concerns European markets, PP is the polymer type with higher demand – over 8 Mtonnes a year, since 2011, according to Plastics Europe (2014). In this sense, a recent study in the Italian coast (Vianello et al., 2013) found the most predominant microplastics to be polyethylene (48%) and polypropylene (38%), in line with the most commonly produced plastic polymers. In this study the lower match was 60% regarding a Rayon fibre.

This work describes values in the range reported by Browne et al., 2011, in a study on the abundances of microplastic particles.

![Fig. 3. Distribution of microplastics by depth for all sampling sites.](image3)

![Fig. 4. Infrared spectrum for sample ALST 119 R3 and comparison with reference spectra for Rayon and Cellulose.](image4)

![Fig. 5. Chemical structure of α-cellulose (Rayon).](image5)
strategies can be developed to minimize marine litter (particularly plastics and microplastics). This measures and strategies should be considered under the MSFD.

Assessing the abundance and distribution of microplastics in the marine environment is challenging owing to the lack of standardization in the sampling and analytical methodologies. Further studies need to be conducted in order to identify distribution patterns and hotspots for microplastic particles in water and sediment samples.

Acknowledgements

The authors would kindly like to thank the support of Mónica Albuquerque and Inês Tojeira from the Portuguese Task Group for the Extension of the Continental Shelf (2013 MrBiS campaign in Algarve), for providing us the subtidal sediment samples; Maria João Melo from the Department of Conservation and Restoration, FCT-UNL, for her support and supervision on the μ-FTIR analysis; Fundação para a Ciência e Tecnologia (FCT) for their financial support through the fellowships reference numbers SFRH/BD/74772/2010; SFRH/BD/74574/2010 and project reference number PTDC/MAR/102677/2008. The work of Jesus Gago was funded by a grant from Xunta de Galicia and co-financed with funds from FSE-Galicia 2007–2013.

in sediments ranging between 2 (in Australia) and 31 (Portugal and the United Kingdom) particles per 250 mL of sediment.

Analysis of polymers using μ-FTIR is described as a definitive method when a good match between sample and reference spectra is obtained. An approach proposed by the TG-ML (Galgani et al., 2013) and followed in this study, is to automatically accept any match >70% similarity, to individually examine matches between 60 and 70% similarity rejecting any samples which do not show clear evidence of peaks corresponding to known synthetic materials and to routinely reject (as being synthetic) any samples which produce spectra with a match < 60%. On the other hand, it could be argued that mismatch between library data and microplastics collected is reflecting that these microplastic particles have suffered degradation, bio fouling, surface adsorption of natural substances of organic and inorganic nature or result from laboratory contamination.

5. Concluding remarks

These results show for the first time the presence of microplastics in coastal sediments from the South of Portugal. Despite the reduced number of samples, it was possible to retrieve 31 microplastics, amongst fibres and fragments. Microplastics were found on 56% of the samples (15 samples over 27). There are few studies that have confirmed the presence of microplastics in coastal low depth sediment samples and the hereby present results tackle this knowledge gap. In this study most of the samples were identified at a depth beneath 20 m.

The vast majority of fibres (81%) were identified as Rayon, a semi synthetic manmade polymer which has been reported in other studies (Lusher et al., 2013), whose use range from hygiene products to clothing. Rayon fibres may be introduced in the marine environment through wastewater effluents, particularly by washing machines (Browne et al., 2011; Woodall et al., 2014).

The remaining microplastics found (19%) were identified as PP. The prevalence of microplastic fibres in all sediment samples studied suggests this contaminant is ubiquitous in coastal sediments.

It is important to note that quantification of microplastics across Europe, is not only relevant for identification of polymer types, abundance and concentrations of microplastics, but also to identify its sources, so that measures and management and planning

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