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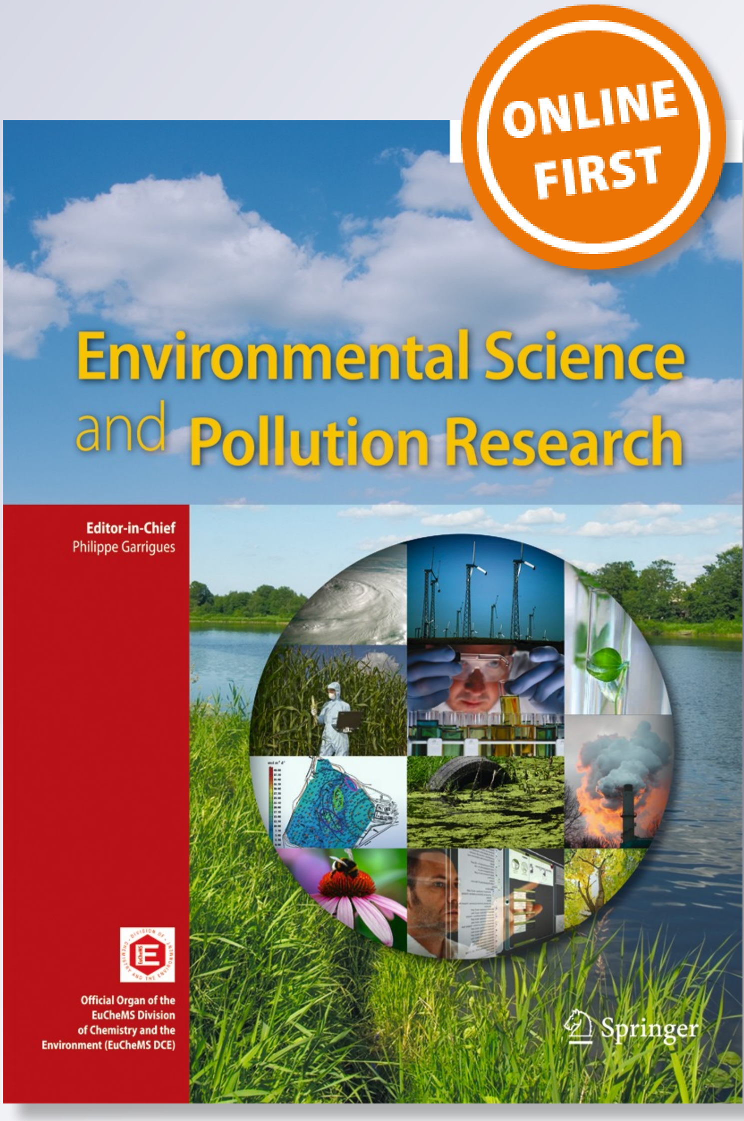

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RESEARCH ARTICLE

The occurrence of microplastic contamination in littoral sediments of the Persian Gulf, Iran

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Abstract Microplastics (MPs; <5 mm) in aquatic environments are an emerging contaminant of concern due to their possible ecological and biological consequences. This study addresses that MP quantification and morphology to assess the abundance, distribution, and polymer types in littoral surface sediments of the Persian Gulf were performed. A two-step method, with precautions taken to avoid possible airborne contamination, was applied to extract MPs from sediments collected at five sites during low tide. MPs were found in 80% of the samples. Across all sites, fiber particles were the most dominate shape (88%), followed by films (11.2%) and fragments (0.8%). There were significant differences in MP particle concentration between sampling sites (p value <0.05). The sediments with the highest numbers of MPs were from sites in the vicinity of highly populated centers and municipal effluent discharges. FTIR analysis showed that polyethylene (PE), nylon, and polyethylene terephthalate (PET) were the most abundant polymer types. More than half of the observed MPs (56%) were in the size category of 1–4.7 mm length, with the remaining particles (44%) being in the size range of 10 μm to <1 mm. Compared to literature data from other regions, intertidal sediments in the Persian Gulf cannot be characterized as a hot spot for MP pollution. The present study could, however, provide useful background information for further investigations and management policies to understand the sources, transport, and potential effects on marine life in the Persian Gulf.

Keywords Microplastic · Coastal pollution · Polymer · FTIR

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Introduction

Marine plastic debris occurs in seas and oceans from the poles to the equator and from remote shorelines to highly populated and industrialized coastlines (Dekiff et al. 2014; Derraik 2002). Pollution of the marine environment is a global phenomenon, and plastic debris presents an increased threat to ecosystems and aquatic organisms according to its durability and persistence (Hidalgo-Ruz et al. 2012; Lusher et al. 2014). Most plastics, such as single-use materials, are discarded within a year of their production (Hopewell et al. 2009), making them the most abundant type of marine debris (Lusher et al. 2014). Since 1950, global plastic production has continued to grow by 9% annually (PlasticsEurope 2013). Once it enters the aquatic environment, large items break down into smaller particles by photolytic, mechanical, and biological degradation (Andrady 2011; Browne et al. 2007). These smaller plastic particles or fibers are generally termed microplastics with a diameter <5 mm (Hidalgo-Ruz et al. 2012). Moreover, plastic debris may also include primary plastic particles produced in

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microscopic sizes including granulates used in personal care products (PCPs), such as cosmetics, washing powders, cleaning agents, or pellets (Fendall and Sewell 2009).

Microplastics (MPs) are distributed throughout all sea compartments including sediments (Alomar et al. 2016). MPs can either float on the sea surface or sink when they become covered with biofilm, ultimately settling into the sediment layer (Wright et al. 2013). MP pollution in the world's oceans has been recently estimated at over five trillion floating particles, corresponding to 250,000 t (Eriksen et al. 2014). Consumption is forecast to increase, with regions like Asia leading the growth. An estimated 80% of plastic in the sea originates from inland sources and is transmitted by rivers to the oceans (Mani et al. 2015). Plastic pollution is the dominant type of anthropogenic debris found throughout the marine environment (Barnes et al. 2009; Eriksen et al. 2013). Plastic debris can physically harm wildlife and enter the food chain with potential human health risks by consumption of contaminated seafood (Browne et al. 2008; Cole et al. 2013; Farrell and Nelson 2013; Lusher et al. 2013; Rochman et al. 2013; Setälä et al. 2014; Vethaak and Leslie 2016). They also have the potential to harm marine biota by alteration of habitats, transport of pathogens/alien species and release of toxic chemicals (Andrady 2011; Mato et al. 2001; Rios et al. 2007).

The objective of the present study was to investigate the abundance, distribution and composition of marine microplastics within intertidal surface sediments along the beaches of the Persian Gulf, Iran. The lack of uniformity across previous studies indicates a need to examine systematically where MPs tend to accumulate across the beach zone. Previously we have reported on MP abundances within the high-tide line (Naji et al. 2017). Here we report on samples obtained at the same five sampling sites but within the low-tide line, and compare those data to abundances within the high-tide line in order to best assess where MPs tend to accumulate.

Materials and methods

Study area

Iran has an extended sea-coast border which is estimated to be about 1770 km on the northern part of the Gulf of Oman and Persian Gulf (Fisher 1968). The Persian Gulf is the third largest gulf in the world (after the Gulf of Mexico and the Hudson Gulf), with a total area of 240,000 km^2 (Naser 2013). In the past few decades, Persian Gulf countries have witnessed major industrial, residential, and tourism development activities (Al-Abdulrazzak et al. 2015, Naser 2013, Sheppard et al. 2010) causing it to be classified among the most anthropogenically impacted areas in the world (Halpern et al. 2008). Being located in a major area for the petroleum industry, oil extraction, and the passage of oil tankers (with an annual estimate of 35,000 tankers crossing the Strait of Hormuz), these activities have a destructive impact on its marine ecosystem (Naser 2013).

The Persian Gulf is a semi-enclosed body of water situated in a subtropical region of the Middle East (Fig. 1). The Gulf is host to some of the most magnificent marine fauna and flora including intertidal mudflats, sea grass, algal beds, mangroves, and coral reefs, some of which are at serious environmental risk (Price et al. 1993; Sale et al. 2011; Sheppard et al. 2010). According to the United Nations Food and Agriculture Organization (FAO), the fishery potential in the Gulf is estimated to be 550,000 t annually or eight times greater than that of the Gulf of Oman (Al-Abdulrazzak et al. 2015; Sale et al. 2011). Intense fishing activities could, thus, be considered a potential source of plastic pollution by fixed and floating fishing gear, as well as discarded or abandoned nets (Aytan et al. 2016). Coastal cities, ports, shipping activities, uncontrolled coastal landfills, and dumping sites along the coast are also important sources of plastic pollution in the area. The origin of marine debris found on the Bandar Abbas, Hormozgan province, beaches is mainly due to beach users' behavior through intentional or accidental dumping. Though minor in comparison with tourism and recreational activities, the second most common source of marine litter was found to be from fisheries activities (Naji et al. 2017; Sarafraz et al. 2016) (Fig. 2).

This area (sampling sites 1, 2, 3, 4, and 5) was chosen for the present study because of its close vicinity to most urbanized and heavily populated area with about one million people living around this area. For example, sites S2 and S3 had the highest values and lie along the vicinity of highly populated centers and municipal effluent discharges, whereas sediments from site S4 contained no MP particles which might be explained by the low anthropogenic influence at this site or local hydrological conditions preventing debris accumulation. The main sources of MP inputs into sites S2 and S3 are urban activities and untreated and/or inadequately treated domestic and industrial sewerage, respectively. The retrieved MPs were likely carried by surface run-off, seasonal river waters, and atmospheric inputs to the sampling sites; however, there is no specific information on atmospheric and municipal effluent discharge from wastewater treatment inputs of MPs in the region. However, you can see discharging of untreated waste water into the Gulf. Untreated and inadequately treated domestic and industrial sewerage are discharging in S2 and S5, respectively.

Recent studies (Mason et al. 2016a, Murphy et al. 2016) indicated that municipal effluent discharge from wastewater treatment works can be a significant contributor of MPs to the aquatic environment owing to microbeads within personal care products, as well as the washing of synthetic clothes (Browne et al. 2011).

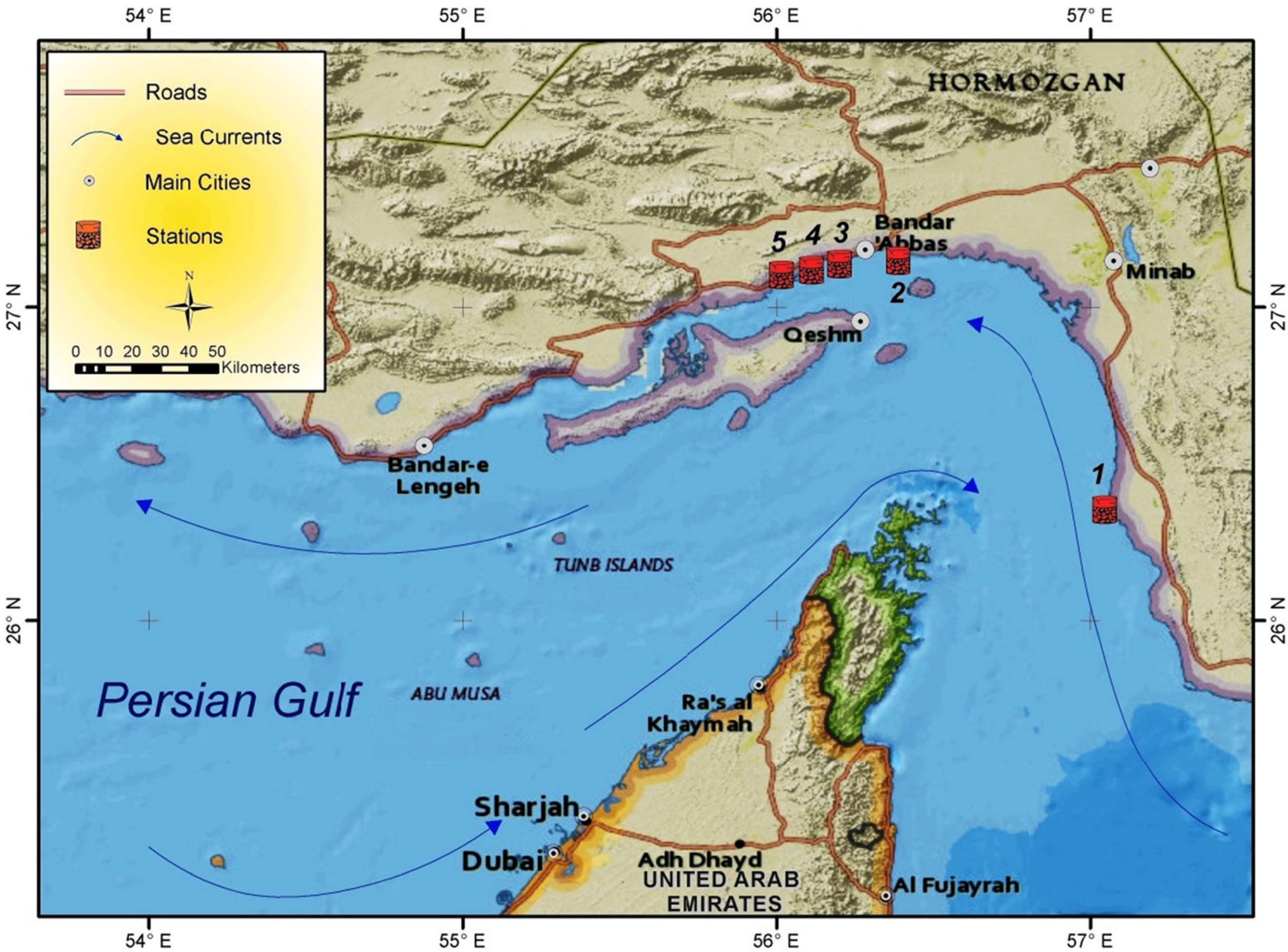


Fig. 1 Map of the sampling area with geographic position of the various sampling sites: *S1* = Khor-e-Azini; *S2* = Khor-e-Yekshabeh; *S3* = Gorsozan; *S4* = Suru; *S5* = Bostanu.

Fig. 2 Example of macroplastic litter items as source of microplastic formation in the Persian Gulf, Hormozgan Province, Iran (photo credit: Dr. A. Naji and GR. Plooijer, 2016)

Sampling collection

The sampling campaign was carried out during January and February 2016. Surface sediments were randomly taken at five sampling sites in the littoral zone during low tide. At each site, three samples were collected. The sites were located in the Hormozgan province on the northern side of Persian Gulf (Fig. 1). The tidal range in the study area varied from about 9 to 10 ft. (2.7 to 3.0 m). The geographical position and characteristics of the sampling sites are shown in Table 1. The longitudes and latitudes of sampling sites were measured by global positioning system (GPS).

Samples were taken from a wooden square frame with side length equal to 1 m. The top 1–2 cm of surface sediment within each 1 m^2 square sampling quadrant was obtained using a stainless steel spoon, yielding a total wet weight of approximately 3 kg for each sample. The spoon was cleaned between samples using sea water and lint-free paper. Sediment samples were preserved in brown glass bottles and transported to the base camp as soon as possible. The bottles were sealed and stored in the laboratory at room temperature for about a week prior to processing.

Sample processing

To separate the MPs from sediments, the recently developed two-step air-induced overflow (AIO) extraction procedure was used (Nuelle et al. 2014). Laboratory setup and method were conducted in accordance with Nuelle et al. (2014), but modifications from the established method are described in Naji et al. (2017). The principle of this method is to use a combination of fluidization of sediments in a lower-density salt solution (NaCl (1.2 g/cm^3)) followed by flotation of MPs in a higher density salt solution (NaI (1.8 g/cm^3)). Extractions were performed without the H_2O_2 "oxidation step," because the samples were not rich in biogenic materials and contained only small quantities of biogenic material. According to Nuelle et al. (2014), the extraction procedure without the oxidation step was shown to have a recovery of >90% for the majority of tested polymer types, and >95% for common polymers including polyethylene (PE), polypropylene (PP), and polyvinyl chloride (PVC).

After homogenizing each sample, 2 kg of wet sediment from each sample (15 samples in total, $n = 3$ per site) was transferred to 500-mL ceramic bowls, which were covered with aluminum foil, and placed in a drying oven at 60 °C until the sample had dried to constant weight. After homogenizing the dried sediment for each sample, 1 kg of dry sediment was utilized for analysis by first sieving through a 4.75-mm stainless steel mesh. Larger MPs were extracted using metal forceps after visual inspection. Suspect particles were further analyzed by FTIR (see below) or verified by a hot needle test (Devriese et al. 2015).

The first extraction step was performed based on the fluidization of the sediments in a saturated sodium chloride (NaCl) (grade 99.5% Darmstadt, Germany) solution (26% weight/weight) with a density of 1.2 g/cm^3. The aim of this first step was to decrease the sediment sample mass for the second density separation step by flotation. Following NaCl flotation, all small particles were collected via filtration through 25-μm stainless steel sieve. Particles were rinsed in distilled water to remove the salts and then dried (60 °C).

In the second MP extraction step, particles were then passed into 500 mL of the sodium iodide (NaI) (grade 99.5%, Darmstadt, Germany) solution (60% weight/weight, with a density of 1.8 g/cm^3), without modification from the method of Nuelle et al. (2014). Within this solution, MPs floated and were exclusively found in the top 200 mL of solution, which was then poured through a vacuum filtration unit fitted with a 0.45-μm nitrocellulose filter (Sartorius Stedim Biotech, Göttingen, Germany). The filter was air-dried and plastics removed for further analysis. MPs were washed in distilled water to remove salts.

Table 1 Geographic position and major characteristics of the sampling sites. All samples collected within the low-tide line

Sites	Name of site	Latitude (N)	Longitude (E)	Description	Main organisms living in each site
S1	Khor-e-Azini	26° 19′ 47.30″	57° 06′ 26.76″	Marine protected area (MPA) mangrove forest.	Mudskipper, *Cerithidea cingulate*, Barnacles, Crabs (*Uca iranica*, *Uca sindensis*, *Scylla serrata*)
S2	Khor-e- Yekshabeh	27° 10′ 39.47″	56° 22′ 11.76″	Nearby municipal area, mangrove forest, and estuary	Mudskipper, *Cerithidea cingulate*, *Crabs* (*Uca iranica*, Uca sindensis)
S3	Gorsozan	26° 10′ 49.67″	56° 17′ 34.45″	In the vicinity of untreated and/or inadequately treated domestic sewerage	Crabs (*Uca iranica*),*Cerithidea cingulate*, *Callista ambunella*
S4	Suru	27° 09′ 19.49″	56° 13′ 53.19″	Urban area	Crabs (*Uca iranica*),*Cerithidea cingulate*, *Callista ambunella*
S5	Bostanu	27° 04′ 58.18″	56° 00′ 27.29″	Industrial area	Crabs (*Uca iranica*), *Cerithidea cingulate*, *Callista ambunella*

Optical analysis of all suspected plastic particles was performed using a stereomicroscope (NOVEL NSZ-810, Ningbo Yongxin Opitics Co., Ltd., Zhejiang, China). Images of suspected particles were taken using a digital camera which was connected to the microscope. The maximum length and width of the MPs were measured using image analysis. A representative subsample of particles that were optically identified as potential plastics was separated using forceps for corroboratory FTIR analysis. Some suspect plastic particles not analyzed by FTIR were verified with a hot needle. The hot point will make the plastic sticky and leave a mark (Devriese et al. 2015). This approach could only be used for the larger micro particles (>1 mm). In total, 68 individual suspected particles were analyzed across all five sites.

FTIR analysis

Suspected MP particles were analyzed using a Bruker (Vertex 70, Germany) Fourier Transform Infrared Spectroscopy (FTIR) to identify the polymer compositions of MPs. FTIR absorption spectra were recorded as an average of 64 scans in the mid-infrared range 4000 to 400/cm at a resolution of 4/cm. The polymer type was identified based on absorption frequencies for specific chemical bond types present in relevant polymer samples.

Quality assurance and quality control

To preclude uncertain contaminations, all laboratory equipment and glass vessels were rinsed two times with double distilled water, left to dry at room temperature within a fume hood, and covered with aluminum foil immediately after drying them. As suggested by Foekema et al. (2013), fibers could be airborne contamination from clothing. Therefore, cotton lab coats, clothing, and gloves were worn at all times during analysis to reduce contamination. The sieve was covered to prevent airborne fibers affecting the sample. All glass vessels and material used for the first time for one sample were covered after each step and cleaned using filtered water before reuse. After filtration, the collected samples were immediately folded and wrapped in aluminum foil. Prior to laboratory analysis, work surfaces were cleaned with alcohol, and hands and forearms scrubbed to prevent contamination from skin, hair, and dirt particles. The workplace for stereomicroscopic analysis was cleaned before opening and analyzing the petri dishes in which MPs were stored.

According to Baldwin et al. (2016), five blank samples in which DI water were stored within sample containers for periods of 3–10 days were processed in a manner consistent with the sediment samples to assess potential contamination from laboratory containers or air. None were found to have any microplastic particulate, indicating that the risk of sample

Table 2 Sediment composition (expressed as % clay, silt and sand, $n = 3$) and mean number of microplastics (particles/kg dry sediment) (±SD, $n = 3$) found in low-tide surface sediments at each sampling site. Mean microplastic particle numbers are provided and then the proportion (in %) found in each step of the air-induced over flow (AIO) extraction procedure; the proportion (in %)of microplastics that were classed as F1 and F2 are presented

Sites	% Sediment type (± SD)			Mean MPs (particles/kg dry sediment) ± SD	F1	F2	Max length (mm)	Min length (mm)	Max width (mm)	Min width (mm)
	Clay <0.05 mm	Silt 0.05–2 mm	Sand >2 mm							
S1	24.0 (5)	67.0 (19)	9 (2)	42.7 ± 5.5	7.69 (18.0)	35.01 (82.0)	2.98	0.82	1.50	0.30
S2	28.6 (7)	46.2 (5)	25.2 (15)	125 ± 25	24.75 (19. 8)	100.25 (80.2)	4.60	3.70	3.55	0.23
S3	18.0 (3)	7.0 (1)	75.0 (22)	103 ± 12.6	13.90 (13.5)	89.10 (86.5)	4.22	1.89	3.05	0.08
S4	16.6 (8)	13.0 (5)	68.4 (16)	n.d.	–	–	–	–	–	–
S5	18.6 (10)	63.0 (22)	18.8 (10)	36.0 ± 7.2	19.00 (52.8)	17.00 (47.2)	2.10	3.20	1.37	0.06

n.d. not detected, *F1* fluidization of the sediments in NaCl, *F2* flotation in a NaI

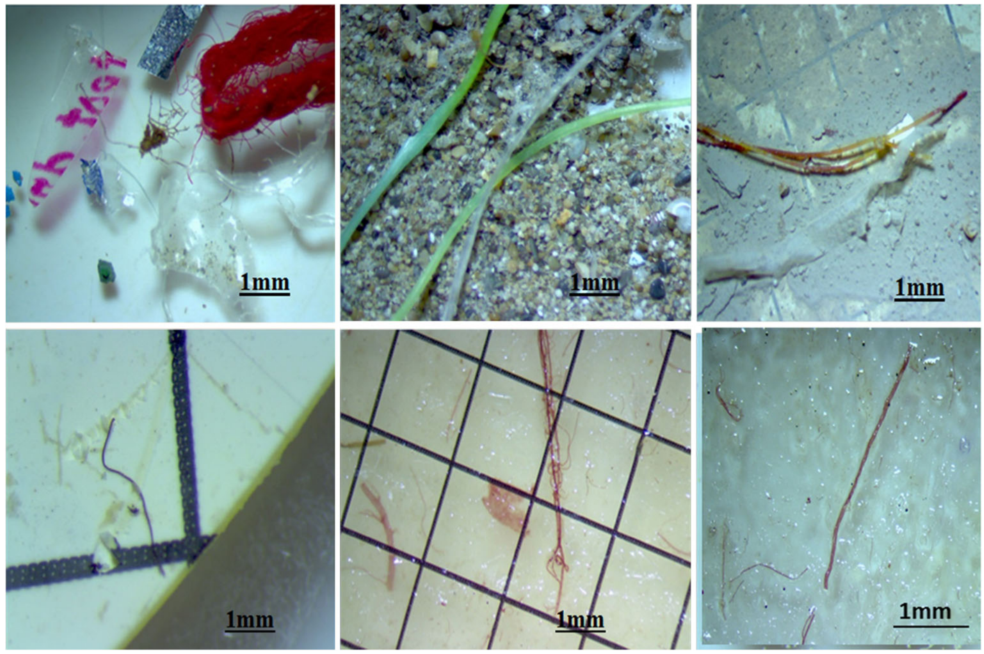


Fig. 3 Examples of microplastics and plankton extracted in surface sediment of the Persian Gulf. Image captured with a stereomicroscope-NOVEL; NSZ-810

contamination from the containers, lab, or processing was negligible.

Statistical analysis

All statistical analyses were computed using Statistical Package for Social Science (version 16.0, SPSS, Inc.), and the graphs were performed with Microsoft Excel 2007 for Windows. Prior to statistical analysis, all data were tested for the basic assumptions for normality and homogeneity of variance. The Kolmogorov-Smirnov test was performed to analyze the normality of the data distribution. One-way analysis of variance (ANOVA) was conducted for multiple group comparisons. A $p < 0.05$ was considered statistically significant.

Results

Occurrence of microplastics

MPs were found in 80% of samples. On average, 26% of the retrieved MPs were extracted during the fluidization of sediment in a saturated sodium chloride (F1), with the remaining 74% obtained in the next step (F2) based on flotation in a sodium iodide (NaI) solution (Table 2). This indicates that the density of ~26% of the extracted MPs is less than 1.2 g/cm^3. A total of 307 particles were identified at all sites from approx. 30 kg dry sediment, with ranges between 0 and 125 particles per kg dry sediment. The average MP concentration across all samples, irrespective of the sampling site, was 61 ± 49 particles per kg dry sediment.

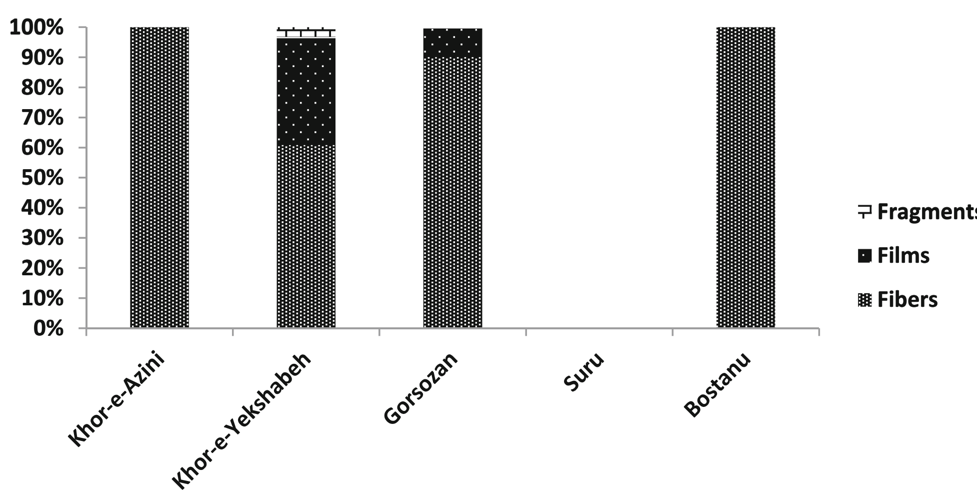


Fig. 4 Relative contribution (in %) of fragments, films, and fibers to total microplastic particle concentrations in surface sediments of Persian Gulf

Table 3 Results of one-way ANOVA analysis for number of MPs in surface sediments of Persian Gulf

Source of variance	df	F	*p*
Occurrence of MPs between stations	3	26.90	0.00

df degrees of freedom, *F* Fisher's constant; *p* value of significance

Identification of polymer types

A total of 68 particles (~22%) were analyzed to identify common polymers from a representative subset of suspected particles recovered from all sites. Of the 68 samples analyzed across all sites, polyethylene terephthalate (PET, $n = 28$, 41%), polyethylene (PE, $n = 21$, 31%), and nylon ($n = 11$, 16%) were the most abundant polymers found. Figure 5 provides representative spectra obtained from extracted MPs for these three polymers. Eight of 68 suspected particles (~11%) could not be identified as common polymeric materials and thus are not considered to be plastic. This result further supports the findings of others as to the importance of spectral analysis for verification of suspected plastic particles.

Discussion

The highest number of MPs in surface sediment was extracted at site S2 (Khor-e-Yekshabeh). The mean concentrations of MPs in the littoral sediments of the Persian Gulf were found to be in descending order: S2> S3> S1> S5> S4, which is consistent with the degree of urbanization in the surrounding areas. The relative contribution of the different types of MPs at each site showed that fibers were the most prominent (average ~88%), followed by plastic films (average ~11.2%) and fragments (average ~0.8%) (Figs. 3 and 4). Fibers were found to be the most common anthropogenic particle type within the samples, though differentiation of synthetic particles from natural cellulose fibers prior to FTIR analysis is known to be problematic because of their similar chemical structure (Lusher et al. 2013; Lusher et al. 2014; Sadri and Thompson 2014). The one-way ANOVA results showed statistically significant ($p < 0.05$) differences among the five sites for MP concentration in littoral sediment of the Persian Gulf (Table 3). The results indicate that MP particle concentrations are significantly ($p < 0.05$) higher at sites S2 and S3 than at other sites.

The highest and smallest lengths (in at least one of its dimensions) among the detected MPs in the study area were 4.6 and 0.82 mm, found at S2 and S1, respectively. The highest and smallest widths were found to be 3.6 and 0.06 mm, both measured at S2 (Table 1).

The highest concentrations of MP particles were found at sites S2 and S3, respectively, and both of these sites are located near recreational fishing sites, as well as construction sites and municipal wastewater discharges. Site S2 with highest concentration is surrounded by mangroves. It has been shown that mangrove soils and roots could trap and immobilize contaminants and that mangroves could function as a purifier of pollutants (Hanum 2014). Intense fishing activities can be considered as a source of nylon by fixed and floating fishing gear, and discarded or abandoned nets in the study area. The high concentration of PET particles in site 3 could be connected with releasing of untreated and/or inadequately treated domestic sewage at this study site. Disposal of municipal wastewater contaminated with fibers from washing clothes was reported as a major source of plastic fibers (Browne et al. 2011).

Identification of polymer types

Our findings are consistent with other studies conducted within open-waters (Besseling et al. 2015; Dekiff et al. 2014; Mason et al. 2016b; Qiu et al. 2015; Thompson et al. 2004; Zbyszewski and Corcoran 2011), as well as production trends. PE is the common produced plastic, being primarily used in packaging (plastic bags, plastic films, containers bottle, etc.). Nylon resins are widely used in the automobile and food packaging industries, and nylon filaments are also used as monofilaments in fishing line. PET is the most common thermoplastic polymer resin of polyester family and is used in fibers for clothing and containers for liquid and foods. All of these polymers indicate an urban origin of this debris.

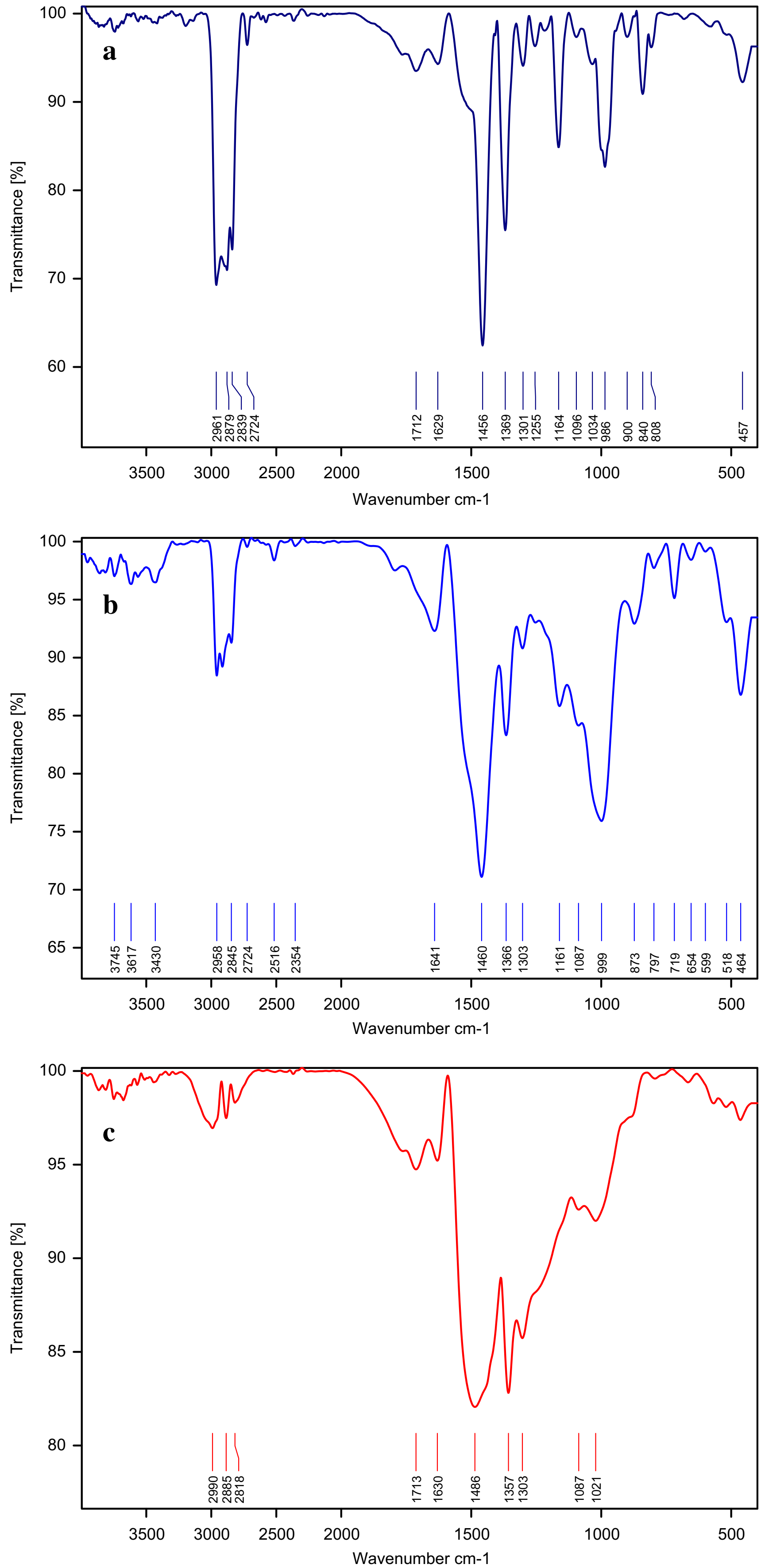
Table 4 Comparison of abundance of MPs (particles per kg dry sediment) low and high tides of same area in Persian Gulf sediments. Size of MPs in both tidal levels was <5 mm

Site	Name of site	Maximal MPs in low tide[a]	Maximal MPs in high tide[b]
S1	Khor-e-Azini	42.7 ± 5.5	2 ± 1
S2	Khor-e-Yekshabeh	125 ± 25	26 ± 6
S3	Gorsozan	103 ± 12.6	122 ± 23
S4	Suru	n.d	14 ± 4
S5	Bostanu	36.0 ± 7.2	1258 ± 291

[a] Present study

[b] Naji et al. 2017

Fig. 5 Identification of microplastic particles extracted from surface sediments of Persian Gulf using FTIR. **a** Polyethylene (PE). **b** Nylon. **c** Polyethylene terephthalate (PET)



Comparison of MPs: high tide vs. low tide

Owing to different sampling and analytical methodologies, as well as an occasionally lack of transparency in lab procedures, a useful comparison of our results with previous studies seems incongruous. Additionally, the majority of previous studies focused on the high tide and reviewed studies show the need to examine systematically where MPs tend to accumulate across the beach zone. We, therefore, decided to compare concentrations of MP particles in low- and high-tide beach sediments for contrast (Table 4). The range of abundances and maximum number of MP particles at low tide were consistently lower than those at high tide except for mangrove sampling sites (S1, S2). As may be expected, our results showed that the concentrations of MPs at sites near highly populated areas were higher at high tide than low tide. The higher levels found in mangroves can be explained by the fact that soils and roots could trap and immobilize plastic debris especially at low tide. However, further studies will be needed to adequately assess the state of distribution of MPs in high and low tides of mangrove sediments.

Conclusions and future research needs

This study presents the first report on the occurrence and spatial distribution of MPs in low-tide surface sediments of the Persian Gulf. The results identified several types of commonly used plastic polymers such as PE, nylon, and PET (Fig. 5). The highest concentrations of MPs were found to be at the beach of S2 and S3 along the vicinity of highly populated centers, municipal effluent discharge, and mangrove forest. We argue to further investigate the Persian Gulf region for the presence and potential impacts of MP waste management, and daily human activities should be controlled in order to reduce the impacts of marine plastic debris in the study area.

The present study provides evidence of MP pollution of the Persian Gulf, and longitudinal studies are required to fully understand the distribution and occurrence of MPs in the area, which are likely to fluctuate both spatially and seasonally.

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