Baseline

Microplastic pollution in the surface sediments collected from Sishili Bay, North Yellow Sea, China

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ABSTRACT

As a new emergence pollutant, microplastic has aroused wide concern from both scholars and the public. In this study, microplastic pollution in surface sediments from 28 stations in Sishili Bay was investigated. The average abundance of microplastics was 499.76 ± 370.07 items/kg (d.w.). Fiber was the majority shape of microplastics (86.37%), followed by film, fragment and pellet. Microplastics < 500 μm accounted for more than half of the total microplastics. Eight polymer types including rayon, PE, PP, PA, PET, PS, PMMA and PU were identified. The main component was rayon (58.41%), followed by PP and PET. The microplastic pollution in surface sediments of Sishili Bay is moderate compared with other studies. Microplastic pollution level in port, sewage outfall, estuary and aquaculture area of Sishili Bay was relatively high, which indicated that microplastic pollution was mainly sourced from river and seawage discharge and maritime activities.

The total global production of plastics has grown from 1.5 million tons in 1950 to 335 million tons in 2016 over the past half century (Plastics Europe, 2017). A study shows that 4.8 million tons of plastic waste enters the ocean each year, and the cumulative quantity of plastic waste available to enter the ocean from land by 2015 is predicted to increase by an order of magnitude (Jambeck et al., 2015). Plastics in the ocean undergo physical degradation, photodegradation and biodegradation into microplastics (plastic fragments smaller than 5 mm in diameter) (Arthur et al., 2009; Law and Thompson, 2014), which are also referred to as secondary microplastics (Auta et al., 2017). Plastic particles manufactured in microscopic sizes are defined as primary microplastics (Cole et al., 2011). Recently, microplastic pollution in the oceans, which represents a growing concern, has been detected around global oceans (Eriksen et al., 2013; do Sul et al., 2013; Collignon et al., 2012; Lusher et al., 2015). Microplastics have been detected in various marine habitats, including the water column, marine sediments, the deep-sea, polar region and sea ice (Law, 2017). In China, microplastics in sediments have been reported in estuaries (Fok and Cheung, 2015; Peng et al., 2017), the Bohai Sea and Yellow Sea (Zhao et al., 2018), and the East China Sea (Zhang et al., 2019). However, the pollution level and characterization of microplastics in nearshore bay areas is rarely reported.

Sishili Bay is located in the North Yellow Sea and borders Yantai, which is an important port city and fishery base in China. Sishili Bay has a mid-latitude monsoon climate: the average annual temperature is 12.6 °C, and the average annual rainfall is approximately 737 mm. The main tidal components in Sishili Bay are semi-diurnal tidal currents. The maximum flow of Sishili Bay is located in the northeast watershed outside Yangma Island with a velocity value of 0.17–0.20 m/s, while the minimum flow rate occurs at the south end of Yangma Island, which is only about 0.04–0.05 m/s. With rapid economic growth, Sishili Bay is under great pressure from human activities, such as maritime shipping, fishing and aquaculture, which have a certain degree of influence on the ecological security of the nearby sea area. Therefore, this study investigates the surface sediments collected from Sishili Bay to reveal the microplastic pollution level through quantification the abundance of microplastics and analysis the characteristics of the size, shape and chemical composition of the microplastics. Moreover, this study will supply the basic data for risk assessment of microplastics in the north Yellow Sea of China.
According to the influence of human activities, this study selects 28 sampling sites in Sishili Bay, which are divided into ZF (Zhifu District), LS (Laishan District) and MP (Muping District) according to different geographical features and urban functions, and which include different types of sea areas such as port, scenic, estuary and aquaculture areas (Fig. 1). The depths of sampling sites ranged from 4 m to 24 m. During cruises in June 2017, the sediments were sampled in triplicate at each sampling site using a stainless steel box sampler and the top layer (depth ~5 cm) was collected with a metal scoop. Each sediment sample with a mass of approximate 250 g was stored in an aluminum foil bag and refrigerated at −20 °C prior to analysis.

A density separation method as conducted in Thompson et al. (2004) was employed to isolate microplastics from sediments with slight adaptations. Briefly, all sediment samples were dried at 60 °C for 72 h to constant weight. 200 mL of saturated sodium chloride solution was added to separate the microplastics from 50 g of sediments using density floatation. Three replicates were completed to increase the recycling rate of microplastics. To reduce interference of organic impurities on subsequent experiments (Nuelle et al., 2014), 5 mL H2O2 (30%) was added to the supernatant to dissolve the biogenic matter. After 24 h of sedimentation, the supernatant was then transferred and filtered over 2.0 μm glass fiber filter paper (Whatman GF/B) under vacuum filtration to retain the microplastics. Finally, filter membranes were collected in sealed petri dishes and air dried for 24 h for further quantitative and qualitative analysis. There were three replicates for each sampling site.

Microplastics were counted and observed by a z-shaped pattern from left to right under a stereomicroscope (Olympus, SZX10, Japan). The microplastic particles were identified with the following criteria: 1) particles have no tissue or organic structures; 2) colored particles are homogenously colored and easy to distinguish; 3) particles are difficult to break using tweezers. Furthermore, all the microplastics were photographed with a stereomicroscope equipped with a camera (Cnptec TP510, Chongqing, China), and the video camera system was used to measure the maximum edge length of the microplastics.

Fourier transform infrared microspectroscopy (μ-FT-IR) was applied to test the polymer types of microplastic. Using a Nicolet™ iN10 infrared microscope (Thermo Fisher Scientific, USA), the randomly selected microplastics from the filters of all the sampling sites were placed onto an ultra-fast motorized stage and measured in the transmittance mode by the MCT detector. The detector spectrum was 4000–650 cm⁻¹, co-adding 128 scans at a resolution of 8 cm⁻¹. The aperture was set at 150 × 150 μm. The spectra were obtained with the OMNIC software (Thermo Scientific, Madison, USA) and compared with the OMNIC polymer spectra library, which matched over 70% with the standard database were acceptable.

To avoid background contamination, all experimental equipments used in the experiment were non-plastic materials. All glass materials and equipments were thoroughly pre-cleaned and rinsed with distilled water three times before and after use. Additionally, all the experimental solutions were adopted after vacuum extraction with 2 μm glass fiber membrane (GF/F Whatman). Operators wore cotton lab coats and nitrile gloves during all experimental procedures to avoid microplastic pollution. Three blank replicate tests were conducted for background correction.

To test the correlation between microplastic abundance and granularity or organic matter (OM) content in sediment, the granularity distribution of the collected sediment was measured with a laser particle size analyzer (BT-9300ST, Bettersize, China), and the organic matter content was determined by loss on ignition.

All statistical analyses were performed using SPSS 16.0 software (SPSS Inc., Chicago, USA). Differences of microplastic abundance between the sampling sites were assessed by non-parametric Kruskal–Wallis tests, followed by pair-wise Mann–Whitney U tests. The correlation between microplastic abundance and the granularity or OM content at each sampling site was tested using Pearson’s method. In all statistical analyses, the significance level was considered as P < 0.05. Maps and tables were produced using ArcGIS (version 10.2.2) and
Microplastics were detected at all stations, and no polymer was found in procedural blanks. In total, 2055 microplastics were separated, with the abundances ranging from 140 to 1873.33 items/kg of dry weight sediment (i.e., d.w.) and an average abundance of 499.76 ± 370.07 items/kg d.w. The distribution of microplastics in sediment samples of Sishili Bay is shown in Fig. 2. In recent years, research on microplastics in marine environment has developed rapidly, but the methods of extracting microplastics from sediments still lack uniform standards. In Table 2, we enumerated published research that used similar extracting methods and quantification units. In comparison with previous studies of microplastic pollution in sediments, the microplastics abundance in surface sediments collected from Sishili Bay was higher than those from mangrove sediments in Singapore and Portuguese coastal sediments (Nor and Obbard, 2014; Frias et al., 2016) and lower than those from North Sea coastal sediments (Leslie et al., 2017) and Canada Halifax Harbor (Mathalon and Hill, 2014). Our study revealed a similar microplastics contamination level with Canada Humber Bay (Corcoran et al., 2015). Therefore, our results suggested that microplastic pollution in the surface sediments collected from Sishili Bay is moderate.

Microplastics particles were detected in all stations, indicating the widespread distribution of microplastics in Sishili Bay. The average abundances of microplastics in ZF, LS and MP were 835.56 ± 736.48, 355.0 ± 214.91 and 479.37 ± 275.32 items/kg d.w., respectively. As shown in Fig. 3, the highest microplastics concentration was found at the ZF1 site, and the MP9 site had the lowest concentration. The microplastic abundance in sediment of the ZF1 site was significantly higher than those of the other sites (P < 0.05). It was reported that microplastic contamination levels were positively correlated with riverine input and human population density (Andrady, 2011; Browne et al., 2011). The ZF1–ZF3 section is located in the Yantai Port, and the ZF1 site is close to the harbor. The Yantai Port, one of the main ports in China, has a cargo handling capacity of 400 million tons per year. A large number of shipping business such as passenger and freight transport might lead to the high regional microplastic contamination level. In the LS section, the highest microplastics abundance was observed at the LS4 site. Because of proximity to Kongtong Island, which is heavily visited by both tourists and local people, the LS4 site exhibited a high microplastics abundance as a result of human activities. Microplastics in the Muping (MP) area were distributed widely but inhomogeneous. The average abundance of microplastics in each section tended to decrease with increased distance from the shore, and the average abundance of microplastics in coastal section reached 670.83 items/kg d.w. Studies have indicated that land input is a significant source of microplastics to the ocean (Browne et al., 2011; Collignon et al., 2012). The MP3 site is located near the Xin’an River estuary, Xin’an wastewater treatment plant and Yangma Island, and the MP7 site is adjacent to sewage outlet. Thus, huge amounts of sanitary and industrial sewage and tourism activities are sources of the high abundance of microplastics. Similarly, the MP5 site might receive a large number of microplastics from the Qinshui River. Moreover, the MP area is an important marine aquaculture area, with a total production of cultured shellfish reaching 50,000 tons per year. The sites MP17 and MP19 are located in scallop culture area, frequent marine activities and extensive use of plastic products such as scallop cages and ropes may lead to the increase of microplastics abundance. Furthermore, microplastics in nearshore area can be transported by waves, tides and currents across the ocean (Zhang, 2017), which results in higher microplastics abundance in some remote regions, such as the MP5–MP11–MP17 and MP7–MP13–MP19 sections.

In this study, there was a moderate correlation between microplastic abundance and OM content in surface sediment (P < 0.05, r = 0.38). Generally, organic matter mainly derived from sedimentation of biomass and external inputs such as river, sewage and aquaculture, and high OM content was found in port, estuary and aquaculture areas. The similar spatial distribution patterns of OM and microplastic indicated that they probably derived from the same source. However, no significant correlation was detected between microplastic abundance and granularity distribution of surface sediments (Supplementary Table S1), which was similar to the results of Browne et al. (2010, 2011).

Typical examples of the four types (fiber, fragments, film and pellet) of microplastics are presented in Fig. 4. As illustrated in Fig. 5, fiber was the most dominant component across all sample types, with a proportion of 86.37%, and only a small amount of microplastics were
fractures (7.83%), films (5.40%) and pellets (0.39%). Fibers made up 86.37% of the total microplastics particles, which was similar to the proportion of samples from Irish coastal sediments (85%) (Martin et al., 2017) and the Bohai Sea and the Yellow Sea surface sediments (93.88%) (Zhao et al., 2018). High percentages of fibers in sediments were also detected in Croatia and Slovenia with a value of 75% and 90%, respectively (Nel and Froneman, 2015; Laglbauer et al., 2014). However, we found that the microfiber abundances of the nearshore sites close to the port, river and sewage outfall were higher than those of the offshore sites (Supplementary Fig. S1), which suggested that microfibers were mainly sourced from land-based sources. It was suggested that the main sources of fiber microplastics were sewages and rivers with fibers generated by washing clothes (Browne et al., 2011; Mishra et al., 2019). Meanwhile, the ropes and fishing nets widely used in aquaculture from nearby shellfish farms were also a possible source (Nor and Obbard, 2014). Thus, we suggested that microfiber pollution might be mainly sourced from sewages, rivers and maritime activities. On the other hand, the higher abundance of film, fragment and pellet microplastics observed at the ZF1 site was likely caused by various maritime activities such as shipping and recreational activities.

The average size of microplastics was 746.84 ± 839.69 μm, ranging from 34.97 to 4983.73 μm. Furthermore, the majority of the microplastics (85.89%) found in this study comprised particles with a diameter of < 1500 μm, and the most common size of microplastics particles was below 500 μm (54.99%). The proportion of microplastics decreased with increasing size (Fig. 6). The numerous small sized particles detected suggested that microplastics were probably from effluent, not only from the breakdown of large plastic items (Yu et al., 2018).

A total number of 228 randomly selected particles were analyzed by

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**Fig. 3.** Microplastic abundances in surface sediments collected from 28 sites of the Sishili Bay. Means in two random groups that do not have the same letter are significantly different at P < 0.05. Vertical bars represent the mean ± S.D. (n = 3).

**Fig. 4.** Different shapes of microplastics in surface sediments collected from the Sishili Bay. A. pellet, B and C. fragments, D and E. fibers, F. film.
μ-FT-IR, and 214 particles were identified as microplastics, with a success rate of microplastics identification of 93.86%. A total number of 8 polymer types were identified (Fig. 7), including rayon, polyethylene (PE), polypropylene (PP), polyamide (PA), polyethylene terephthalate (PET), polystyrene (PS), poly (methyl methacrylate) (PMMA) and polyurethane (PU), which indicated that the sources of microplastics were very broad. The number of each microplastic polymer type is shown in Table 1. Of all identified samples, the most common polymer type of microplastics was rayon, accounting for 58.41%, which was consistent with the conclusion of sediments from Southern Portuguese, Bohai Sea and a deep-sea area (Frias et al., 2016; Zhao et al., 2018; Woodall et al., 2014). As a fiber composed of regenerated cellulose, rayon is mainly used in personal hygiene products and textiles (Kauffman, 1993). Therefore, laundry and sanitary wastewater could be the origin of rayon in the ocean (Browne et al., 2011; Woodall et al., 2014). With relatively high percentage of PP (17.76%) and PET (14.95%) microplastics, our result was similar to that observed in sediments from the Arctic deep-sea (Bergmann et al., 2017). In general, PP is widely used in fishing tools and electrical appliances, and PET is mostly used to make electronic devices. In addition, PP and PET are increasingly used in the production of garments, nonwovens, carpets and ropes (Park et al., 2004). Accordingly, land-based sources and maritime activities are probably the main sources of microplastics in surface sediments collected from Sishili Bay.

In summary, microplastics were widespread in surface sediments collected from Sishili Bay. The average microplastics abundance was 499.76 ± 370.07 items/kg d.w., which was at a moderate level compared with similar studies across the world. Microfibers accounted for a dominant proportion of microplastics, and the majority of the particles had a diameter of < 1500 μm. In addition, the most common polymer types observed in sediments were rayon, PP and PET. Our study speculated that land-based sources including river and sewage discharge and maritime activities are probably the main sources of microplastics in surface sediments of Sishili Bay. Our results could supply basic data for environmental risk assessment of microplastics in the
Table 1
Abundance, percentage and density of microplastics samples in surface sediments collected from Sishili Bay.

| Type of polymers | Number | %     | Density g/cm³ | Library                  |
|------------------|--------|-------|---------------|-------------------------|
| Rayon            | 125    | 58.41 | 1.5           | Hummel Polymer Sample Library |
| Polypropylene    | 38     | 17.76 | 0.92          | HR Nicolet Sample Library |
| Polystyrene tetrahydrofuran | 32      | 14.95 | 1.38          | Cross Sections Wizard |
| Polyethylene     | 12     | 5.61  | 0.95          | Polymer Laminate Films   |
| Poly (methyl methacrylate) | 3        | 1.40  | 1.18          | Hummel Polymer Sample Library |
| Polystyrene      | 2      | 0.93  | 1.05          | Hummel Polymer Sample Library |
| Polyamide        | 1      | 0.47  | 1.15          | Hummel Polymer Sample Library |
| Polyurethane     | 1      | 0.47  | 1.05          | HR Nicolet Sample Library |
| Total            | 214    | 100   | /             | /                       |

North Yellow Sea, and further investigation and surveillance of microplastics pollution are needed in China.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.marpolbul.2019.02.021.

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