Microplastics in abiotic compartments of a hypersaline lacustrine ecosystem

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Abstract

The study of microplastics (MPs) in inland water bodies has been growing recently. However, there is still insufficient knowledge of the status of MPs in lacustrine ecosystems, especially in saline lakes. To date, most studies have been conducted on sediment, water and biological compartments of lakes. In this study, for the first time, the status of MPs in abiotic compartments of the saline Maharloo lake in Iran, including surface sediment, lake salt, sludge, lake water and wastewater, is evaluated. A total of 742 MPs, mainly clear, fibrous MPs ranging from 50 to 250 µm and composed of polypropylene and polyethylene terephthalate, were identified in 33 samples. Mean MPs concentrations in solid samples were greater than in liquid samples: the highest levels (51.7 MPs/kg) were found in sludge, and lowest levels, in lake salt (10.4 MPs/kg). The highest MP levels were found in the northwest of the lake where there are wastewater effluents from urban, industrial and agricultural activities discharging into the lake. Interrelationship assessments of MPs with hierarchical cluster analysis suggested that differences in the distribution of MPs with different physical properties in the Maharloo lake are greatly affected by weathering processes and proximity to contamination hotspots. The results of this study revealed that the widespread occurrence of MPs in the Maharloo lake greatly originates from potential plastic sources in urban areas of Shiraz metropolis and its industrial zone in the vicinity of the study area. MPs, then, via surface runoffs, especially wastewater inflows, disperse into the lake.

Keywords: Fibre; Sediment; Water; Salt; Wastewater; Maharloo lake

1. Introduction

Petroleum-based polymers have important presence in our societies because of their physical and chemical characteristics including low density, durability and corrosion resistance. Global plastics production reached almost 360 million tonnes in 2018 (PlasticsEurope, 2019). Plastics have widespread use and great stability, and both factors contribute to their persistence in the environment (Zhang et al., 2020a).

MPs (1 μm − 5mm) move from terrestrial to aquatic ecosystems with different pathways, among which wind, runoff and treated or untreated wastewater discharges are important (Vendel et al., 2017; Cheung et al., 2018). Rivers constitute main pathways of microplastics to the sea (Li et al., 2020), and their sediments can be a sink for MPs, especially in their headwaters (Drummond et al., 2022). MPs can also enter aquatic ecosystems as a consequence of in-situ anthropogenic activities such as fishing or the destruction of plastic parts of ships and boats(Nematollahi et al., 2021). From rivers, MPs can transfer to groundwater, reservoirs, lakes and the sea. However, the flow dynamics and accumulation of MPs in rivers will be unlike other types of surface water due to their different hyporheic exchange processes (Drummond et al., 2022). Eventually, MPs will reach the sea, where MP levels in the pelagic areas are predicted to reach four times current levels by 2060 according to numerical models (Isobe et al., 2019).

MPs in water, plants and those resuspended from accumulations in aquatic sediments may be ingested and cause unfavourable health impacts to aquatic living organisms, including reduced growth rate and reproductive ability, suffocation or restrictions of movement (Alimba et al., 2019). MPs can also act as a vector for toxic chemicals and microorganisms (Koelmans et al., 2016). MPs can accumulate harmful chemicals from water and living organisms. The ingested MPs can contribute to the transfer of the contained harmful substances to the organisms (Koelmans et al., 2016) however the desorption and bioavailability of the sorbed substances is still unclear.

Aquatic biota may carry the ingested MPs for a long time, and subsequently, some MPs may be transferred up in the food chain (Jambeck et al., 2015).

Numerous recent studies have investigated MP occurrence in aquatic environmental compartments (e.g., Savoca et al., 2021; Cera et al., 2022; Soltani et al., 2022). Enclosed water bodies such as lakes can sequestrate more MPs compared to flowing water (Imhof et al., 2013; Free et al., 2014) and hence lakes can give a better indication of long term pollution levels. Nonetheless, a limited number of studies have been conducted in lacustrine environments (e.g., Vaughan et al., 2017; Liang et al., 2021). Saline lakes have been scarcely considered and the salinity of such environments could affect the distribution of MPs in the water column. Increased salinity can lead MPs to become suspended nearer to the surface, precisely their flotation following the addition of salt to aqueous samples is used during their separation for analysis (Nematollahi et al. 2020). In Iran, environmental assessments on MPs contamination have been mostly carried out in the southern Caspian sea (e.g., Rasta et al., 2020, Abadi et al., 2021) and Iranian sectors of the Persian Gulf (e.g., Naji et al., 2017; Nabizadeh et al., 2019). To date, MP pollution has comprehensively not been studied in different compartments of lacustrine ecosystems.

The saline lake Maharloo may be the most important sink of contaminants from Shiraz metropolis and peripheral areas. It is mainly recharged by the seasonal Khoshk river, flowing from central urban areas of Shiraz. The growing rate of urbanization and industrialization of Shiraz in the last decades, and also intense agricultural activity in the vicinity of the Maharloo area, have led to having poorly treated municipal, industrial and agricultural wastewater discharging towards the lake (Forghani et al., 2009). Thus, a study on MP occurrence in Maharloo will give new relevant information on the fate of MP pollution in a saline lake, and environmental information on the ecological state of the Maharloo lake and its surroundings. This study will make possible the identification of MP hotspots that should be remediated.

2. Materials and methods

2.1. Description of the study site

Maharloo is a seasonal inland saline lake situated in the longitude of 52º 48' and latitude of 29º 28' 23 km in the Southeast of Shiraz (Fig. 1). The closest city to the study area is Shiraz. Shiraz has 1,565,572 inhabitants and population density of 248 persons/km. It is the fifth most populated city in the country according to the 2015 census. The lake has an average width and length of 11 and 31 km, respectively, and it covers 250 km^2. In the wet season, Maharloo has a maximum water depth of 3 m. Its mean annual evaporation and precipitation are 2572 mm and 344 mm, respectively (Forghani et al., 2009). The lake is noticeably influenced by water recharge: it leads to 1 m increase in water depth, resulting in 80 Km^2 increase in the lake surface area (Tajabadi et al., 2018). The study area has semiarid weather with an annual average temperature of 17 °C (Eini et al., 2019).

2.2. Sampling, sample preparation and analysis

Sampling was carried out from 16 stations (displayed in Fig. 1) during the wet season in November 2019. Sampling stations were evenly spread around the lake and some were close to probable pollution hotspots including canoeing docks (S6), villages (S11, S12, S15), recreational areas (S5, S6, S14), estuaries (S1-S4, S16), industrial sites (e.g., a salt mining plant) (S7) and remote areas (S8-10). A total of 33 samples including, 12 surface sediment (S4-S7 and S9-S16), 8 lake salt (S6-

S11, S14, S16), 7 lake water (S4-S7 and S14-S16), 3 wastewater (S1-S3) and 3 sludge (S1-S3) samples were collected. Sludge was collected from industrial and urban wastewater. In this study, sludge refers to sewage materials removed from urban and industrial treatment plants after secondary treatment. These are directed by channels to the lake. Due to a slope reduction of the path when these channels enter the lake bed, they settle in the northwest side of the lake. Sediment refers to terrestrial material transported to the lake by surface runoffs, rivers, atmospheric deposition, and also chemical deposition from substances dissolved in water which subsequently sediment with time.

At each sampling sites, surface sediment, lake salt and sludge (1 kg, 0 – 20 cm); lake water (3 l) and wastewater (3 l) were collected. Water collection was carried out from the surface till up to 30 cm deep. Composite sediment and salt samples, including a mixture of 4 subsamples, were sampled using stainless steel shovel. They were kept packed in aluminium foil and labelled. Composite bulk water samples, including 3 bulk water subsamples, were collected and stored in glass bottles (pre-cleaned with filtered distilled water), sealed with aluminium-coated caps and labelled. Samples were transferred to the lab for subsequent treatments.

Sample preparation and treatment were carried out as reported elsewhere (Nematollahi et al., 2020). Briefly, sediment samples were dried at room temperature in a cleanroom. Dried samples were homogenized while disaggregating sediment clumps by pressing them softly using a porcelain pestle and mortar. Following, each sample (100 g) was passed through a stainless steel sieve (5 mm cut-off), transferred to glass beakers and covered with aluminium foil. The treatment of the sediment sample included the oxidation of the sediment organic matter (OM) by adding about 100 ml of 30% hydrogen peroxide (H_2O_2) until the completion of the oxidation reaction (~7 days). Unreacted H_2O_2 in the beakers was removed from the samples in a sand bath at 85 °C for 4 h, and then the sediment samples were washed with pre-filtered water and dried in the sand bath. In this study, 2 μm pore size S&S filter papers (blue band, grade 589/3) were used to filter water and reagents. Such oxidative conditions also cause important decomposition of OM (>87% degradation of organic matter from soil and sludge was achieved with H_2O_2 at 70 °C (Hurley et al 2018)). However, some degradation of polymers was also observed under such conditions and it was recommended to keep the temperature <40 °C for safety and preserving the integrity of MPs (Hurley et al 2018).

MPs were extracted from sediment samples using floatation with $ZnCl_2$ solution (1.7 g cm^{-3}). The $ZnCl_2$ solution (100ml) was added to each beaker containing sediments, shaken for 5 min at 400 rpm and let to to settle for 24 h. With this step, the MPs from the solid samples went to the liquid phase. The liquid phase was separated, transferred to falcon tubes and centrifuged (4000 rpm for 5 minutes). Following, MPs were recovered from the supernatants with filtration supported with a vacuum pump.

The filter papers used were left to dry at room temperature in pre-cleaned sterilized cabinets. Overall, the extraction process of every sediment sample with $ZnCl_2$ was carried out three times. Ultimately, the residual content on the filter papers was transferred to glass Petri dishes for further analysis using a hairbrush made of natural fibres. The protocol for treating sludge samples was the same as for sediment samples. To extract MPs from salt, first, the salt crystals were crushed using agate mortar and pestle. Following, each sample (200 g) was dissolved in pre-filtered deionized water (2 l), and 30 % H_2O_2 was added to the solution for 12 h to digest OM (Liebezeit et al., 2012). Finally, MPs from the salt samples, and also from the surface water samples, were extracted following the same protocol that the procedure followed for the extraction of MPs from the supernatant obtained from sediment samples.

MPs identification and counting were performed under an optical microscope (Carl-Zeiss, Oberkochen/West Germany, 40X optical zoom) aided by an insulin needle and ImageJ software. Based on physical characteristics, MPs were classified into shape (fibre, sheet, fragment and bead), color (black-grey, blue-green, yellow-orange, red-pink and white-transparent) and size/length (L) ($1000 \leq L < 5000$ µm, $500 \leq L < 1000$ µm, $250 \leq L < 500$ µm, $100 \leq L < 250$ µm and 50 µm $\leq L < 100$ µm). The minimum MP size that could be counted was 50 µm.

Representative MPs (2 % of the total number of extracted MPs) with different physical characteristics were separated and mounted onto double-adhesive copper tape stripes using an insulin needle. Their morphology and elemental composition were determined using a Scanning Electron Microscopy (SEM) equipped with an Energy Dispersive X-ray Microanalyzer (EDS) (TESCAN Vega 3, Czech Republic) after copper adhesive tape stripes containing MP particles were coated with gold. A confocal Raman microscope (Lab Ram HR Evolution, Horiba Japan) was used to identify the polymer type of the representative MPs. MPs on copper adhesive tape stripes were mounted on microscope glass slides without gold coating. The excitation source of Raman was a laser irradiating at 785 nm. Raman's detection was in the range of 400−800 cm^{-1}. The identity of the polymer/s making up the MP particles was found by comparing the experimental spectra to the Horiba internal spectral database (KnowltAll®). The acquisition software used was HORBIA Scientific's LabSpec 6.

A number of quality controls were applied to assure the validity of laboratory works. In order to prevent MP contamination, all treatments and analyses were performed in a clean room and cotton gloves and laboratory coats (free from plastic) were used during the experiments. Also, glassware was pre-washed with phosphate-free soap, double-rinsed with pre-filtered distilled water, left for 24 hours in 10% HNO_3, re-rinsed with the same type of water and dried in a "clean" environment at room temperature. Benches were thoroughly cleaned using ethanol 70% before any analysis of the samples. All chemical solutions and reagents used in the study, such as H_2O_2 and $ZnCl_2$, were pre-filtered to remove possible MP particles. In addition, during experimental works, an empty container (blank sample) was left open in the laboratory for four weeks to monitor airborne MPs pollution status (control blank sample). The analysis of the control blank sample confirmed the absence of unwanted MP contamination in the laboratory. Furthermore, to safeguard the complete transfer of MPs from the filter papers to Petri dishes, the swept filter papers were re-examined with optical microscopy. Blanks (n = 2) and a blind sample were examined to evaluate the accuracy of spectroscopic instruments.

2.3. Data processing

To plot maps and display spatial distribution of the data, ArcGIS 10.3 was used. SPSS version 22 was applied to perform statistical analyses. Statistical Shapiro-Wilk (S–W) test was used to examine whether there was a normal distribution of the data in the samples. The homogeneity of variances was also tested and they were not homogeneous ($p < 0.05$). Statistical Kruskal–Wallis (K–W) H-test was applied to find significant relationships in the abundance of MPs between various media (sediment, salt, sludge, lake water and wastewater). Hierarchical cluster analysis (HCA) was applied using Ward’s method to find interrelationships between MPs properties within sampling sites.

3. Results and discussion

This research examines the MP pollution in the Maharloo lake, its distribution and possible origin. The information extracted from the study will help to establish the state of the lake and identify measures to mitigate the pollution in the area and protect it.

3.1. MPs distribution and contamination

The investigation of the occurrence of MPs across sampling sites (16) found a differentiated distribution of MPs ($p < 0.05$), determined with Kruskal-Wallis H test (Table 1). The number of MPs reported in Table 1 is affected by the size of the samples analysed. The MP levels reported were not corrected by their extraction recoveries because of the challenge of having representative MPs from those in the environment for an accurate assessment. However, the sample treatment method used presented consistent high recoveries for MPs (>95%) when the assessment was done with broad composition, sizes and densities. These were assessed elsewere (Imholf et al., 2012).

However, when reporting results in concentrations, sludge is the fraction most contaminated followed by wastewater. Salt constitutes the least contaminated fraction of MPs. Based on Shapiro-Wilk test, MPs had a non-normal distribution within the samples ($p < 0.05$). This can be caused by several contamination hotspots that lead to uneven distribution of MPs in the environment, including elevated MPs concentrations in a number of sampling sites.

Spatial distribution of MPs abundance within different media is shown in Fig 2. The highest levels of MPs were found in S16 (35 MPs kg^{-1}), followed by S7 (33 MPs kg^{-1}), S12 (31 MPs kg^{-1}) and S4 (29 MPs kg^{-1}) in the sediment samples; in S10 (12 MPs kg^{-1}), followed by S16 (11 MPs kg^{-1}) and S14 (11 MPs kg^{-1}) in lake salt samples; and in S4 (30 MPs l^{-1}) followed by S16 (29 MPs l^{-1}) in lake water samples. Hence, the greatest concentrations of MPs in surface sediment and lake water samples were found in the North West of the lake, that is where wastewater from urban, industrial and agricultural activities discharges into the lake. Furthermore, all sludge and wastewater samples in this part of the lake reveal high contamination of MPs, which reflects the influence of greywaters. Overall, this sector of the lake contained 53 % of MPs from four samples (S1, S2, S3, S16). In general, MP contamination derived from wastewater discharged decreased with distance from the source, though some sites far from the discharge point may be affected by other anthropogenic sources, specifically including entertaining activities on the coast and boat riding at the east of the lake. Elevated concentrations of MPs in S7 and S12 (sediment sites) have likely been due to the proximity to a commercial salt production factory and Baharan residential areas, respectively. The latter can contribute to the release of plastic litter that will eventually degrade into MPs. Salt pools in the lake are exploited to supply salt for the production of soda ash in a petrochemical plant and thus can disperse plastic particles into the water. Wastewater from the commercial salt production factory enters the Maharlo lake without sanitary treatment. Baharan village, with 2178 inhabitants, does not have a sanitary treatment plantand its sewage is directly transferred to the lake via a waterway. Human sewage is usually the result of bathing and washing clothes, which is one of the main sources of fibrous microplastics.

With reference to the MP pollution of the salt, all salt samples showed a relatively similar concentration of MPs, i.e., the number of MPs in all sites were approximately the same. The lowest concentration of MPs in salt from the lake was found in S8: where there are layers due to evaporation of lake water, and hence salt could not be contaminated by the wastewater outlet.

The presence of MPs in aquatic environments can arise from coastal debris, industrial pollution, wastewater treatment plants, surface runoffs and tributaries, unmanaged waste dumping and landfilling and atmospheric deposition (Nerland et al., 2014; Crawford and Quinn, 2017;

Magnusson et al., 2016). MPs distribution maps (Figure 2) reveal that proximity to contamination hotspots is a factor in distributing MPs into Maharloo lake. Specifically, samples taken from wastewater inflows with the highest contamination levels confirm that off-situ anthropogenic activities influence releasing MPs in the lake. Previous studies have indicated that MPs in inland water bodies originate from urban, industrial and agricultural inflows and surface runoffs (Baldwin et al., 2020; Dong et al., 2021). In addition, atmospheric transport and dry/wet deposition of MPs, such as synthetic fibres, favour the spread of MPs in the environment (Allen et al., 2019; Szewc et al., 2021). Specifically that the dominant wind direction (southeasterly) is similar to the decreasing trend in the concentration of MPs towards the east of the lake. In a recent study in the vicinity of the study area, Nematollahi et al. (2022) suggested that atmospheric deposition is a crucial factor in transporting MPs towards the south of Shiraz, where Maharloo lake may receive loads of MPs. This assessment was based on Shiraz's topography, where, dominant southeasterly winds in the study area facilitate MPs transfer from urban areas of Shiraz metropolis and its industrial park, right in the vicinity of the study area, towards Maharloo lake in the southeast of Shiraz. Our findings are in line with recent studies (e.g., Vaughan et al., 2017; Hendrickson et al., 2018; Dong et al., 2021) which have indicated the role of surface runoffs and greywaters inflows on MPs occurrence in lakes. In Maharloo lake, land use around the area; severe evaporation and water level decline leading to the presence of vast salt flats around the lake in the dry seasons; in-situ anthropogenic sources such as population density and recreational activities (e.g., boat riding) are not of greater importance than surface runoffs and greywaters inflows, which is proved based on MPs levels within the sampling sites.

3.2. MPs physical properties

Representative MP particles detected in the lake are shown in Fig. 3. They were examined with optical microscopy. Pie diagrams (Fig. 4) illustrate percentage of MPs characteristics and concentration in each environmental compartment of the lake. In addition, spatial distribution of MPs characteristics within the sampling sites is shown in Fig. 5.

3.2.1. Shape

Fibrous particles were the main shape of MPs in each environmental compartment of the lake (Fig. 4), followed by sheets and fragments in surface sediment, sludge, lake water and wastewater, and fragments in lake salt. On the whole, fibres comprised 80 % of MPs found in all compartments of the lake, while sheets, fragments and beads constituted the remaining 12, 6 and 0.4 %, respectively. Recent studies represented fibres as dominant shape of MPs in lakes (e.g., Xia et al., 2020; Cox et al., 2021). Predominance of fibrous MPs over other shapes may be referred to their higher production, e.g., 9 million tonnes in 2016 (Carr, 2017), their high release per se as MP particles, such as microfibres released from textiles (Belzagui et al., 2019) and easy identification because of their relatively large size (Allen et al., 2019). Presence of fibres in surface water bodies is speculated to considerably originate from wastewaters and surface runoffs (Browne et al., 2011; Mason et al., 2016), also from atmospheric deposition (Dris et al., 2016). Given the higher levels of MPs in sludge and wastewater, also in sites in the northwest of the study area (Fig. 2), it is expected that the wastewater inflow of the lake is the most important source. This has led to circulating very higher levels of fibres across the lake compared with other MP shapes (Fig. 5). The occurrence of fibres in the lacustrine ecosystems is largely attributed to urban origins such as washing machines and wastewaters (Dusauny et al., 2021). Hence, fibres in the wastewater inflow of Maharloo lake are expected to mainly originate from potential pollution sources in the vicinity of the lake, including urban wastewaters of Shiraz metropolis and effluents from Shiraz industrial zone. Textiles are a prevalent source of fibres in urban areas that can easily be released from clothing and soft furniture (e.g., carpet and curtain) during washing and enter the urban wastewater system, also they directly can be transported to remote areas via wind currents (Nematollahi et al., 2022). For instance, a recent study reported that laundries can release around 6 million fibres per 5 kg wash into urban wastewater (Rodrigues et al., 2018). In another study by Browne et al. (2011), around 1,900 MP particles were released into urban wastewater during laundry washing. Wastewater treatment plants and other industrial units in the Shiraz Industrial zone in the vicinity of Maharloo lake are also other potential sources emitting fibres into the lake. This has recently been implied in studies carried out on the important role of wastewater treatment plants in releasing fibres into the environment (e.g., Bitter and Lackner, 2020; Wolff et al., 2021).

Shiraz metropolis and its industrial area has a two-stage acoustic aeration activated sludge (EAAS) treatment plant system. In the first stage, large solid objects are separated from the incoming wastewater by metal meshes, and in the next stage, wastewater is purified using air blowers from aerobic and anaerobic bacteria. In the last phase, the treated effluent is released to the Maharlo lake. Since there is no specific mechanism to remove plastic particles present in the wastewater from these treatment plants, the majority of MPs may enter the lake.

3.2.2. Colour

MPs showed a variety of colours, though white-transparent (31 %) MPs were dominant in all compartments, followed by blue-green (24 %), black-grey (20.5 %), red-pink (18.5 %) and yellow-orange (6 %) MPs (Fig. 4). Predominance of clear-coloured MPs in Mharloo lake is in line with those found in other lakes around the world (e.g., Wang et al., 2018; Baldwin et al., 2020; Xia et al., 2020). Among abiotic compartments of the lake, clear (white-transparent) MPs were dominant in sediment (48 %) and wastewater (47.5 %) samples, while black-grey MPs dominated lake water (29 %) and sludge (37 %) samples. In lake salt samples, the difference between dominant and other colours was slight, though blue-green (26.5 %) and black-grey (25 %) MPs had higher proportion than other ones. Spatial distribution of MPs colour indicates that most sampling sites in the east of the lake comprise higher proportion of white-colour and blue-green MPs, while sites in the west constitute a higher contribution of black-grey and white-transparent MPs compared to other colours (Fig. 5). A reason for colour diversity of MPs in the environment arises from various plastic sources (Fahrenfeld et al., 2019) such as textiles, health and beauty products and packaging and plastics industries (Karimi et al., 2018). The colour is widely used in the manufacture of plastic products to promote their attractiveness (Xu et al., 2020). Nonetheless, the identification of MPs origin regarding their colour might occasionally be imprecise, i.e., the colour is impermanent and may undergo photobleaching processes (Wagner and Lambert, 2018). Prevalence of white-transparent MPs in the environment may be an indicator of prolonged exposure to the sun, leading to their photobleaching (Weber and Opp, 2020) and that lake compartments are sinks for MPs. Clear colours may also propose their sources from disposable plastic materials (e.g., plastic bags). In addition, digesting organic matter by acid while sample treatment may lead to discolouration of MP particles and formation of clear fibres (Pfeiffer and Fischer, 2020). Nematollahi et al. (2022) represented that clear MP particles in the dust of Shiraz could originate from abundant applications of disposable plastic products in the commercial and residential sectors of the city, which subsequently could through surface runoffs and atmospheric deposition transport to the southern parts (e.g., Maharloo lake). The presence of colourful MPs in the lake may originate from very resistant, consumable plastic products as suggested elsewhere (Andrady, 2017; Eo et al., 2019), such as worn constructive materials. In addition, presence of colourful MPs in Maharloo lake may reflect low effect of photobleaching, and hence they have been relatively new.

3.2.3. Size

The MPs found had varied sizes, however those with the smallest range size were dominant in all samples and the abundance decreased with an increase in particle size (50 μm ≤ L ≤ 100 μm: 42.9 %, 100 μm ≤ L ≤ 250 μm: 30.9 %, 250 μm ≤ L ≤ 500 μm: 20.6 %, 500 μm ≤ L ≤ 1000 μm: 20.6 % and L ≥ 1000 μm: 9.9 %). Only in the lake water samples, MPs with 100 - 250 μm range had greater abundance than the rest, while the smallest range size was dominant in other compartments. The dominance of the smaller-sized MPs in this research is in line with recent studies in aquatic environments (e.g., Yang et al., 2019; Szewc et al., 2021). Spatial distribution of MPs illustrates predominance of smaller MPs across the lake (Fig. 5). In the Maharloo lake, this may be derived from small plastic materials in a variety of daily used products, but they are also likely affected by the degradation of larger plastics sourced from Shiraz and its industrial park while having been transported into the lake. The MPs with smaller size are potentially more dangerous to aquatic biota than larger MPs. This is in part, potentially, due to having greater penetration into tissues, adsorption of toxic chemicals (Xu et al., 2020) and can be intaken easily (Li et al., 2020).

3.3. Interrelation of MPs properties and spatial distribution

The interrelation between MPs properties (shape, colour and size) and their spatial distribution was investigated using hierarchical cluster analysis (HCA) and spatial distribution maps (Fig. 6). The HCA comprised three clusters (C1, C2 and C3) and thus the lake was divided into three zones:

1) northwestern (C1), 2) central (C2): and 3) eastern zones (C3). Based on the shape, fibres were dominant in all three zones of the lake, although there was an increase in the concentration of fragments from zone 1 to 3. This can be due to the more intense effects of weathering on MPs towards zone 3, such as high degradation and fragmentation during MPs transport from zone 1 (contamination hotspot) towards zone 3 via lake currents, also high photobleaching due to the evaporation and higher proportion of settled MPs exposed to sunlight in zone 3. The original colour of the primary plastic could also have been light or white. MPs larger than 1000 µm had greater abundance in zone 1 than in other zones (see Figs 5 and 6). This is well correlated with MPs size because weathering processes (e.g., photodecomposition) can contribute to breaking down larger particles to smaller ones towards zone 3. It should be noted that the elevated number of MPs in S14 due to proximity to the residential area (Baharan village), and littering from it, has led this station to fall in zone 1.

In terms of colour, zone 1 had higher percentage of black-grey MPs. On the contrary, zone 3 presented higher percentage of white-transparent MPs than other zones (Figs 5 and 6). Prolonged photobleaching in zone 3 may be causing abundance of clear MPs in the eastern parts of the lake. Light colour in the primary plastics generating MPs could also become white-transparent MPs.

Interrelationship assessments indicated that differences in the distribution of MPs with different physical properties in Maharloo lake are greatly affected by weathering processes and proximity to the contamination hotspots.

3.4. MPs weathering

MPs weathering was evaluated by analysis of fifteen representative MP particles with different shapes, colours and sizes (Fig. 7). Weathering signs were observed in most MP particles, including cracks and irregular edges in sheets and fragments (Fig. 7 c and e) and groves in fibres (Fig. 7 b, d and 7). This implies that most particles have been secondary MPs. Despite this, a small number of samples (e.g., Fig. 7a) also included primary MPs with smooth surfaces and regular and sharp edges, reflecting their recent entrance into the lake. Surface morphology of investigated MPs indicated that the degree of weathering in fragments was more intense than in other MP shapes in Maharloo (Fig. 7 e). Weathered MPs facilitate the sorption of harmful organic and inorganic chemicals (Kowalski et al., 2016; Su et al., 2019). The desorption of some of these chemicals from weatherded MPs may occur in living organisms, however the contribution from the ingestion of contaminated MPs appears lower compared to the ingestion contaminated pray (Koalmans et al., 2016).

3.5. Chemical composition

Chemical composition of MPs was investigated by analysis of elemental content and polymer type of 15 representative MPs of different physical characteristics (see Table 2). MPs constituted a variety of major and minor elements. Plastic nature of MPs was proved by a high percentage of C and O, and to a lesser extent N as a major constituent of hydrocarbons, in the analyzed particles. MPs also comprised minor amounts of Al, Si, Pb, Cl, Zn and Cu. The presence of Al and Si as major constituents in the structure of silicates, such as clays, probably reveals that MPs act as a vector to adsorb silicate minerals onto their surface (Nematollahi et al., 2022). A number of major and trace elements (as additives) are generally used in the plastic structure to acquire specific characteristics. For instance, Pb and Cu act as pigments in plastics and paints (Brokbartold et al., 2012; Ogilo et al., 2017). Pb is also used to increase plastic density (Bolgar et al., 2015). Si and Al increase durability of plastics (Bolgar et al., 2015). In addition, elemental constituents of MPs may also come from salt particles adsorbed to MPs surfaces (e.g., zinc chloride salts) and or various chemicals used during sample treatment (Nematollahi et al., 2022). Weathering of MPs can lead to the release of their additives and adsorbed chemicals into the lake ecosystem and threat aquatic biota.

The most prevalent polymers identified by Raman in all compartments of the lake were PP (46.5 %), PET (33.5 %) and PS (20 %) (see Table 2 and Fig. 7). The same polymeric compound of MPs was also found in similar previous studies (e.g., Feng et al., 2021; Dusaucy et al., 2021). PP and PS are broadly used in disposable plastic materials, packaging materials and reusable bags (Barrows et al., 2018; Zhang et al., 2020b). PET is widely applied in the structure of fibres used in textiles, but also in beverage bottles and packaging materials (Gong et al., 2018). MPs found in the sediment, sludge and wastewater samples were mostly constituted PET and PP, while PS was more prevalent in MPs found in lake water and lake salt samples. Several factors may control variations in polymer type of MPs between abiotic compartments of the lake, mainly including potential origins, forms and polymer density of MPs, also watershed properties (Dusaucy et al., 2021). Sheet MPs were made of PET and PP, while fragments were made of PP and PS. Fibres were found from a variety of polymers. In this project, the main polymers making fibres were PET and PP.

The frequency of fibres made of PP and PET in Shiraz was found to arise from synthetic textiles as the main origin for MPs (Nematollahi et al., 2022). Moreover, presence of multiple polymers in the structure of MP itself reflects that MP is are made of of co-polymers such as PET-PS and PS-

PP. Remarkably, MPs of the same colours had different chemical compositions. This may arise from different additives that act as pigments in the structure of MPs.

3.6. MPs in the study area and other similar locations

The MPs status in the abiotic compartments of Maharloo lake is compiled in Table S1. MPs levels in surface sediments, lake salt, sludge and wastewater of the study area were lower than in other locations, except in the Caspian sea (Nematollahi et al., 2020). In addition, the water in Maharloo lake contained moderate concentrations of MPs compared to other locations. In the majority of the studies, and here, fibres were the major shape of MPs. A variety of polymers were detected in lacustrine ecosystems, though PE, PET, PP and PS were the most prevalent polymers. In addition, PP and PET were the most abundant polymers in Maharloo lake. Overall, the differences in the status of MPs in Maharloo lake and other regions can be ascribed to origins distributing MPs to the aquatic ecosystems, different land uses, wind currents, waste management and population density. In addition, microscopic and analytical factors, such as the number of representative samples analysed and the working range of microscopes, also precision of the analyst and the instrument, can influence the results obtained from different studies.

4. Conclusion

This study assessed the widespread occurrence of MPs in Maharloo lake, which was mostly derived from potential plastic sources from peripheral urban and industrial areas. This suggested Maharloo lake as an important sink for MPs. The results indicated that wastewater streams remarkably control MPs dispersion derived from Shiraz metropolis and its industrial park into the lake. Indeed, the abundance of MPs decreased from the west to the east of the lake, which is also supported by dominant wind direction. MPs in abiotic compartments of the lake vary in terms of concentration, physical properties and chemical composition. Clear MPs composed of PP and PET, with 50 to 250 μm in length, had the highest contribution to the MP contamination in the lake. Spatial distribution of MPs physical properties indicated higher proportion of white-colour, larger-sized MPs in the west of the lake, while dominant shape (fibre) was the same across the lake.

Different chemical compositions and physical properties of the investigated MPs evidence that they may arise from a variety of sources. However, the predominance of fibre MPs proposed that synthetic textiles may have had the largest proportion of the MP contamination in the study area.

The results obtained from this research can be a baseline for the MPs status in saline lacustrine ecosystems in future studies. Nonetheless, further studies are needed to evaluate the circulation of MPs between abiotic compartments of saline lakes. In addition, a prospective study on the MPs occurrence in living organisms of saline lakes can highlight the importance of these ecosystems as a sink of MPs. Seasonal sampling of the lake compartments, also upstreams, atmospheric dust and potential sources in remote areas favour identifying possible sources of MPs in the lake

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Caption of Figures:

Fig. 1 Sampling sites in the Maharloo lake. The wind direction in Shiraz and maps showing the location of Maharloo lake in the Fars province.

Fig. 2 Spatial distribution map of MPs concentration (item/L) in **a)** sediment and sludge, **b)** lake salt and **c)** lake water and wastewater sites.

Fig. 3. A variety of MP particle detected under binocular optical microscope (40× optical zoom) with different physical properties; a) fibres, b) fragments, c) elongated sheets, d) hexagonal sheets and e) plastic particles (>1000 μm) detected in the field.

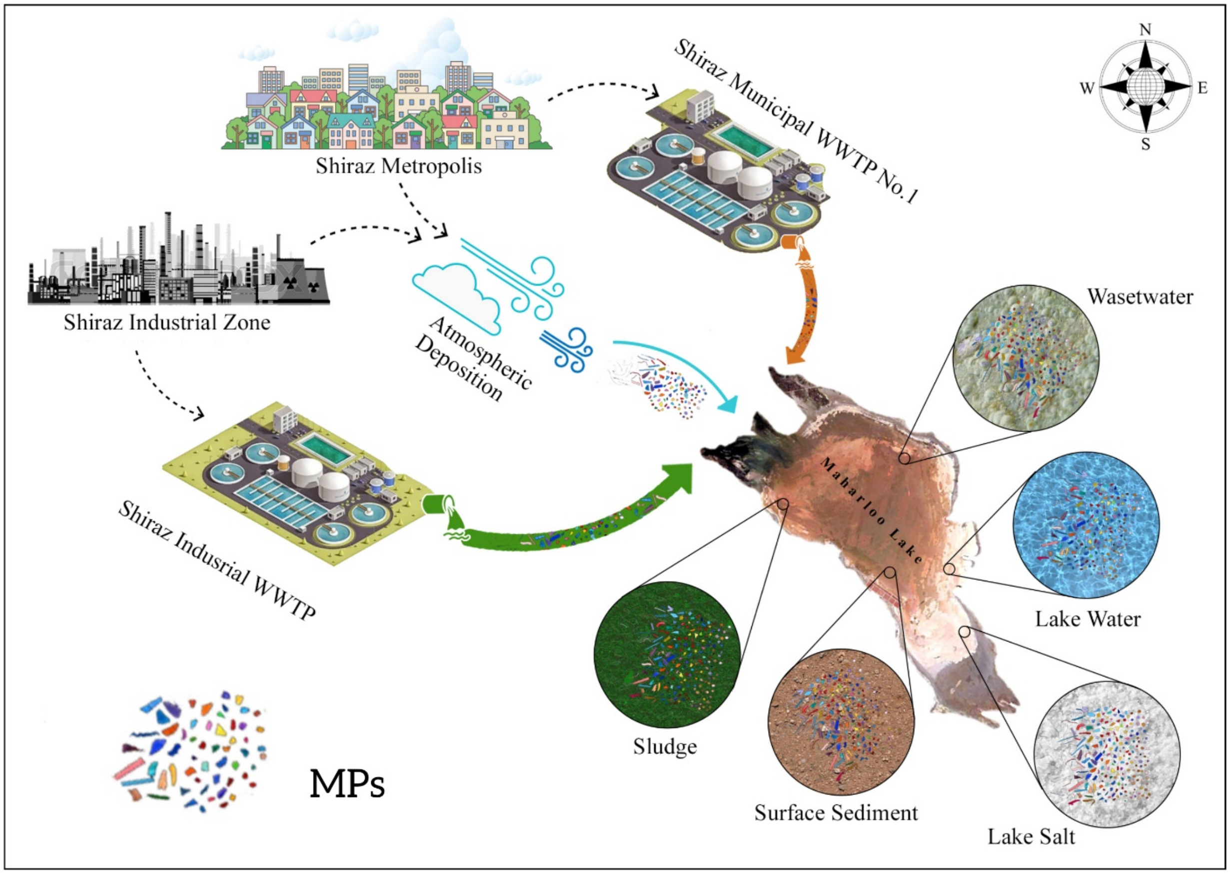
Fig. 4. Percentage of physical properties (shape, colour and size) and concentration of MPs in abiotic compartments in Maharloo lake.

Fig. 5. Spatial distribution of MPs physical properties within sampling sites.

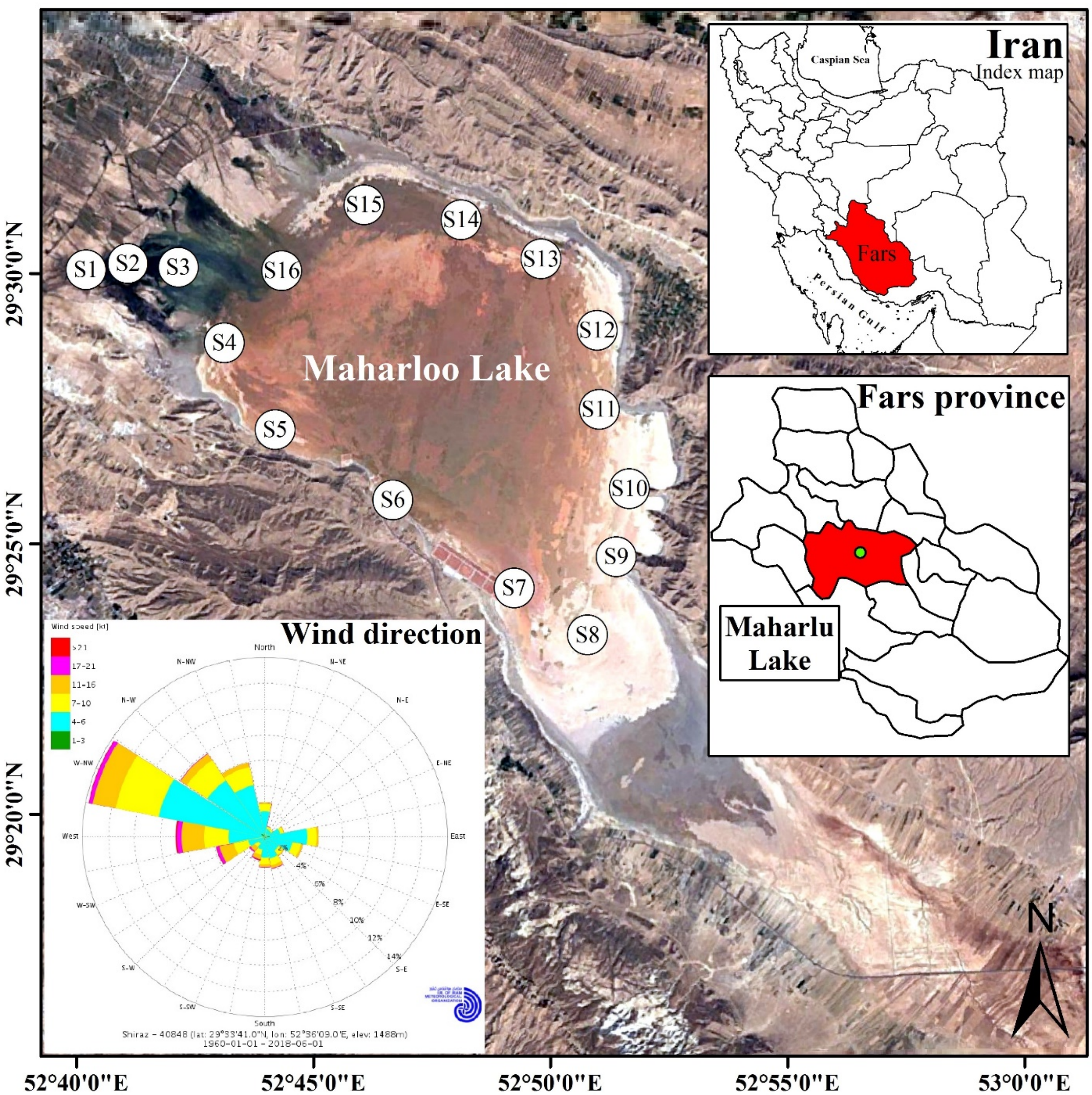
Fig. 6. Zoning (1-3) of MPs properties including **a)** shape, **b)** size and **c)** colour in Maharloo lake based on hierarchical cluster analysis

Fig. 7. SEM, optical microscopy images and Raman spectra of selected MPs in Maharloo lake; **a)** a primary blue hexagonal sheet, **b**) a fairly weathered red fibre, **c)** a moderately weathered yellow hexagonal sheet, **d)** a highly weathered blue fibre, **e)** a highly weathered blue fragment and **f)** a secondary blue fibre

Graphical abstract



Figures1


Iran
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Fars province
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Maharloo Lake
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8%
10%
12%
14%
Shiraz - 40848 (lat: 29°33'41.0"N, lon: 52°36'09.0'E, elev: 1488m)
1960-01-01 - 2018-06-01
N
29°30'0"N
29°25'0"N
29°20'0"N
52°40'0"E
52°45'0"E
52°50'0"E
52°55'0"E
53°0'0"E

Figure 2

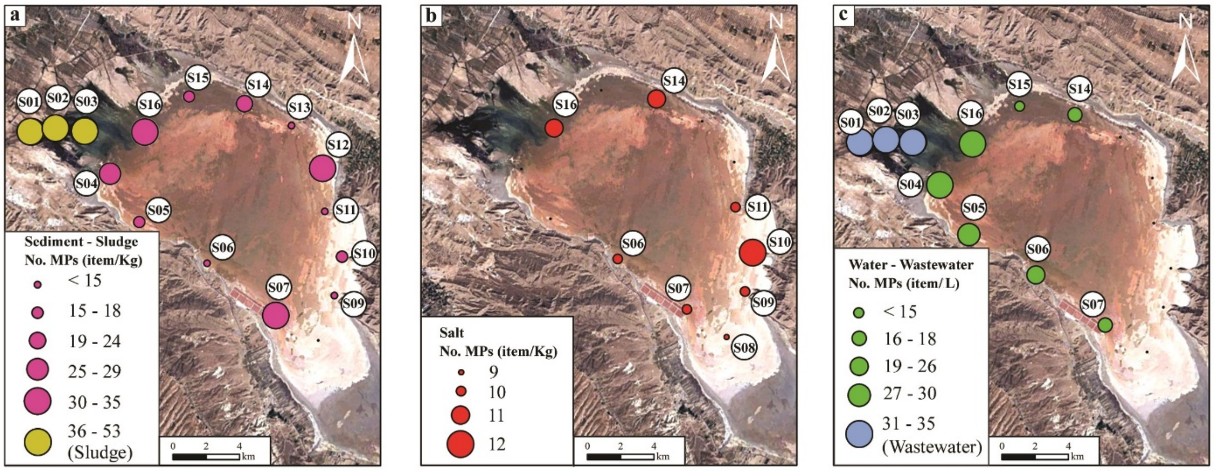

a
S01
S02
S03
S04
S05
S06
S07
S09
S10
S11
S12
S13
S14
S15
S16
Sediment - Sludge
No. MPs (item/Kg)
< 15
15 - 18
19 - 24
25 - 29
30 - 35
36 - 53
(Sludge)
0 2 4 km
b
S06
S07
S08
S09
S10
S11
S14
S16
Salt
No. MPs (item/Kg)
9
10
11
12
0 2 4 km
c
S01
S02
S03
S04
S05
S06
S07
S14
S15
S16
Water - Wastewater
No. MPs (item/L)
< 15
16 - 18
19 - 26
27 - 30
31 - 35
(Wastewater)
0 2 4 km

Figure 3

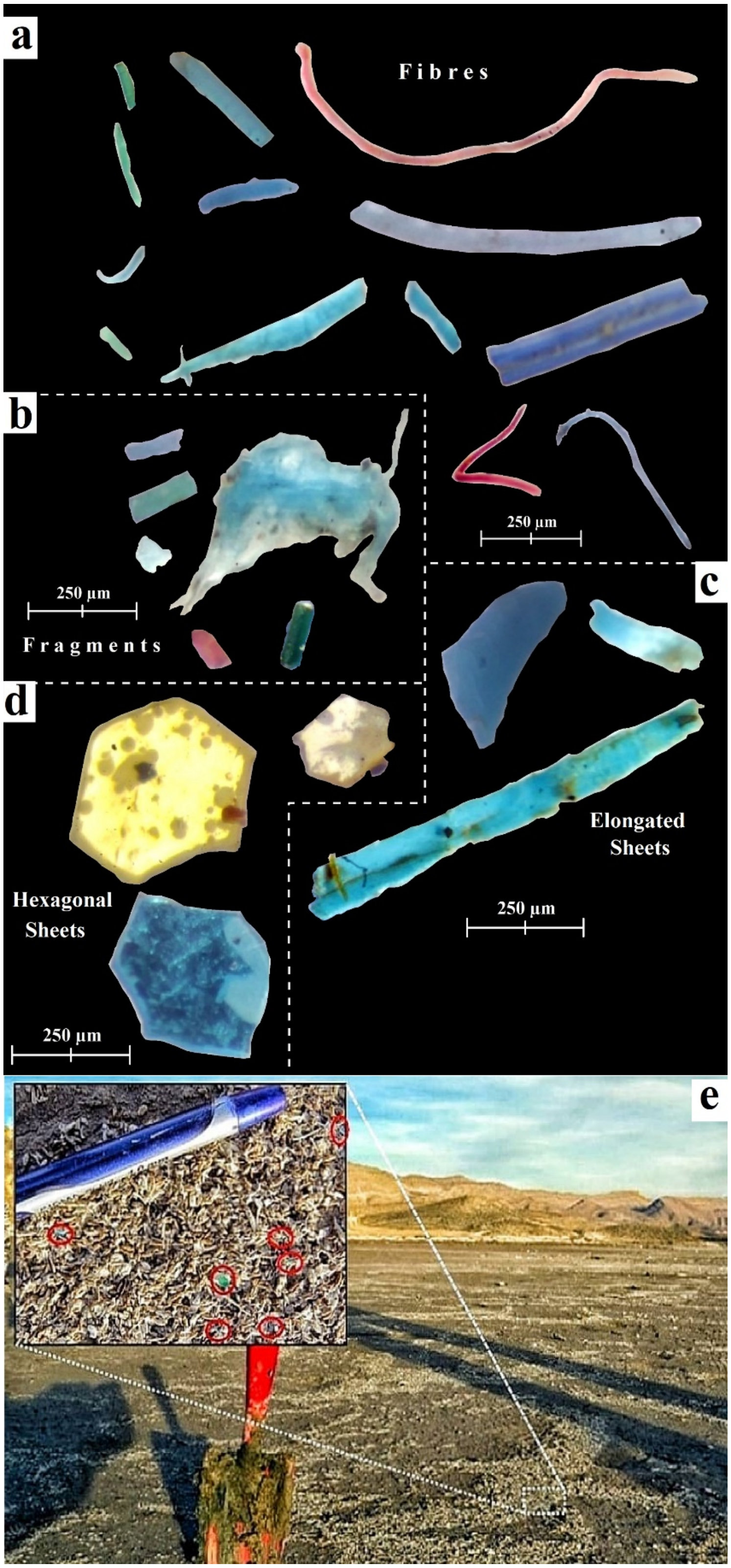

a
Fibres
b
250 µm
Fragments
250 µm
c
d
Elongated Sheets
250 µm
Hexagonal Sheets
250 µm
e

Figure 4

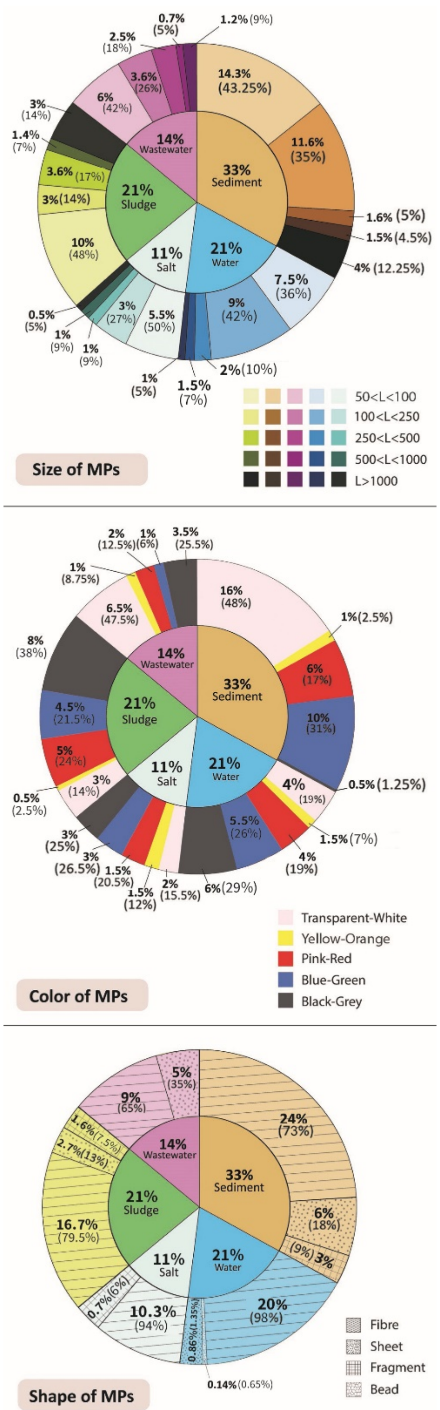
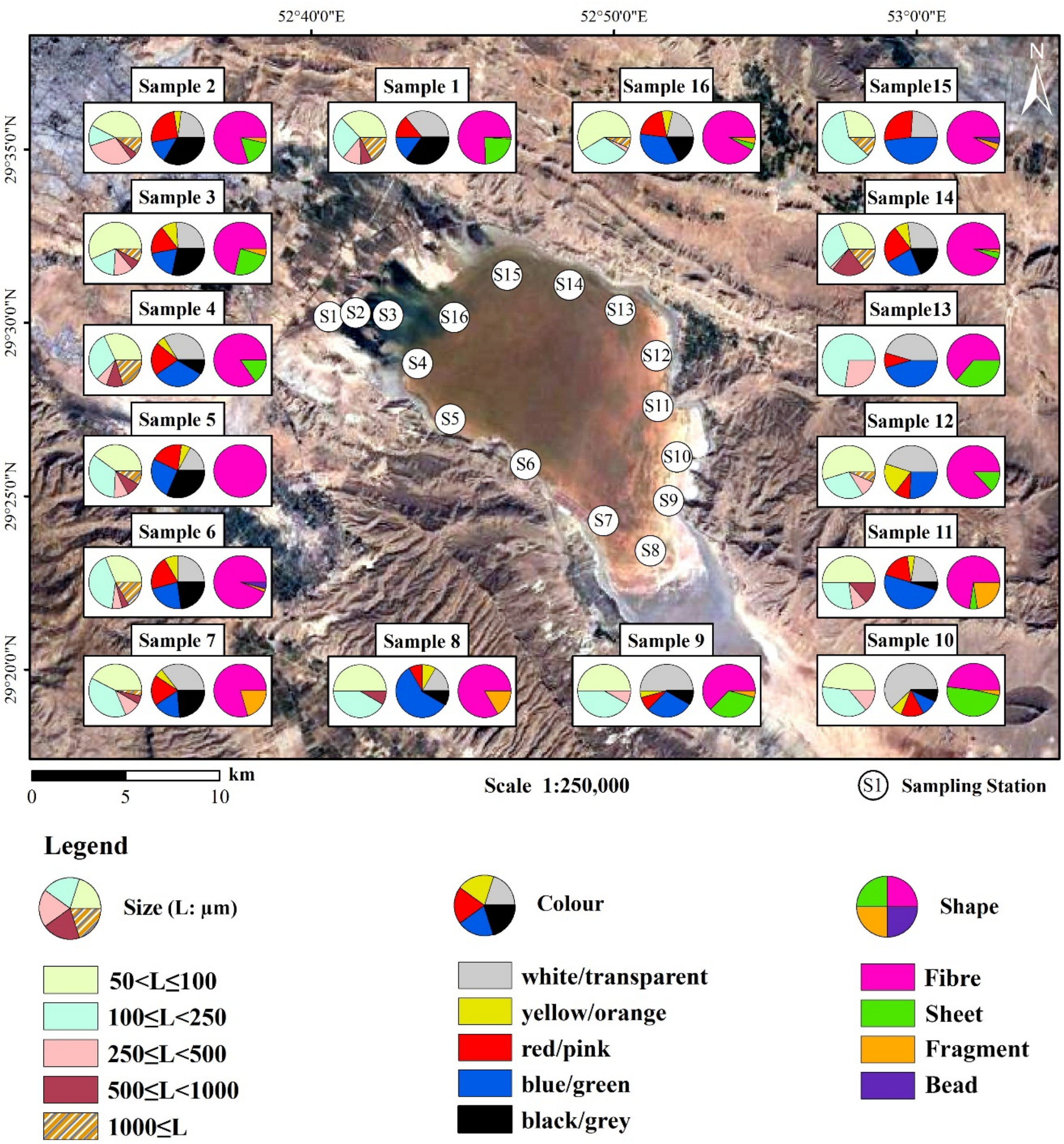

Size of MPs
33% Sediment
21% Water
11% Salt
21% Sludge
14% Wastewater
14.3% (43.25%)
11.6% (35%)
1.6% (5%)
1.5% (4.5%)
4% (12.25%)
7.5% (36%)
9% (42%)
2% (10%)
1.5% (7%)
1% (5%)
5.5% (50%)
3% (27%)
1% (9%)
1% (9%)
0.5% (5%)
10% (48%)
3% (14%)
3.6% (17%)
1.4% (7%)
3% (14%)
6% (42%)
3.6% (26%)
2.5% (18%)
0.7% (5%)
1.2% (9%)
50<L<100
100<L<250
250<L<500
500<L<1000
L>1000
Color of MPs
16% (48%)
1% (2.5%)
6% (17%)
10% (31%)
0.5% (1.25%)
4% (19%)
1.5% (7%)
4% (19%)
5.5% (26%)
6% (29%)
2% (15.5%)
1.5% (12%)
1.5% (20.5%)
3% (26.5%)
3% (25%)
0.5% (2.5%)
3% (14%)
5% (24%)
4.5% (21.5%)
8% (38%)
6.5% (47.5%)
1% (8.75%)
2% (12.5%)
1% (6%)
3.5% (25.5%)
Transparent-White
Yellow-Orange
Pink-Red
Blue-Green
Black-Grey
Shape of MPs
24% (73%)
6% (18%)
(9%) 3%
20% (98%)
0.86% (1.35%)
0.14% (0.65%)
10.3% (94%)
0.7% (6%)
16.7% (79.5%)
2.7% (13%)
1.6% (7.5%)
9% (65%)
5% (35%)
Fibre
Sheet
Fragment
Bead

Figure 5



52°40'0"E
52°50'0"E
53°0'0"E
29°35'0"N
29°30'0"N
29°25'0"N
29°20'0"N
N
Sample 2
Sample 1
Sample 16
Sample15
Sample 3
Sample 14
Sample 4
Sample13
Sample 5
Sample 12
Sample 6
Sample 11
Sample 7
Sample 8
Sample 9
Sample 10
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11
S12
S13
S14
S15
S16
km
0
5
10
Scale 1:250,000
S1 Sampling Station
Legend
Size (L: μm)
Colour
Shape
50<L≤100
100≤L<250
250≤L<500
500≤L<1000
1000≤L
white/transparent
yellow/orange
red/pink
blue/green
black/grey
Fibre
Sheet
Fragment
Bead

Figure 6

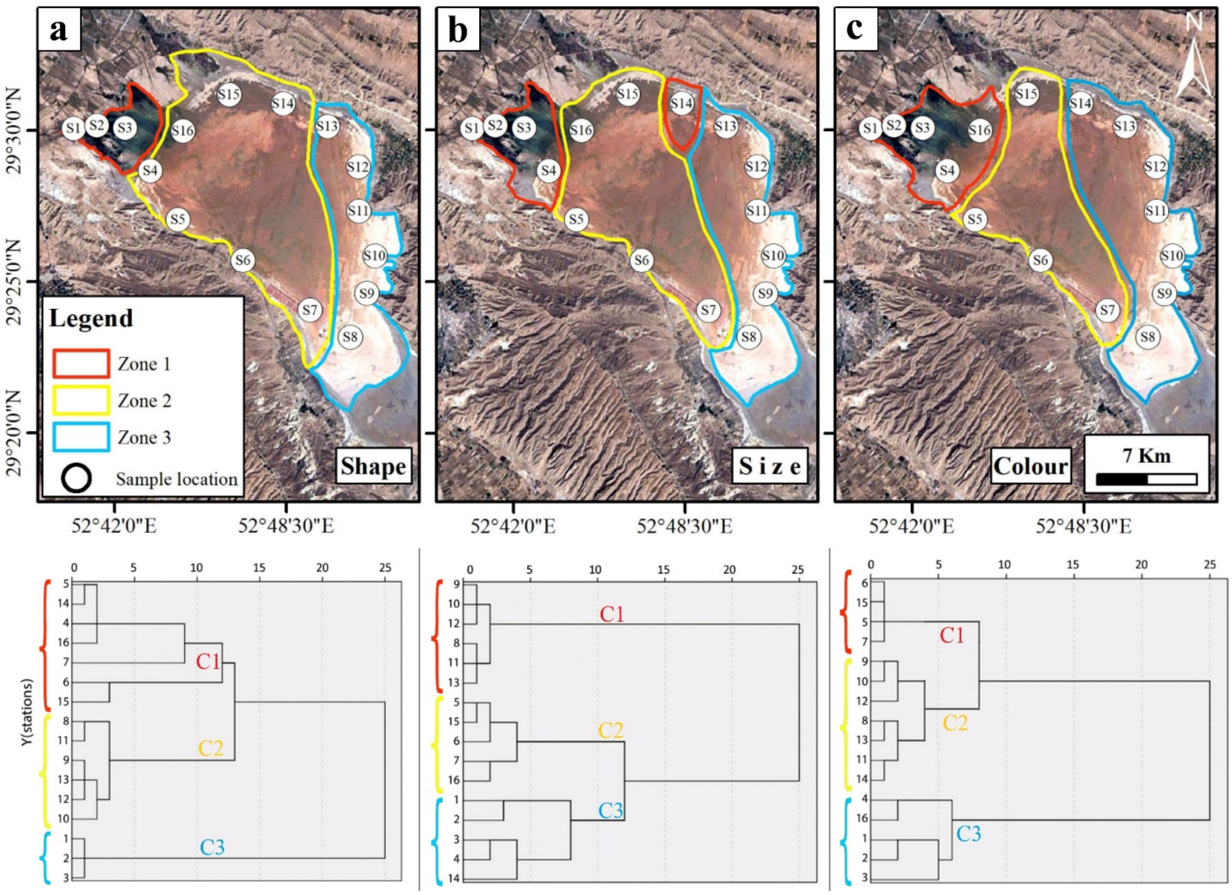
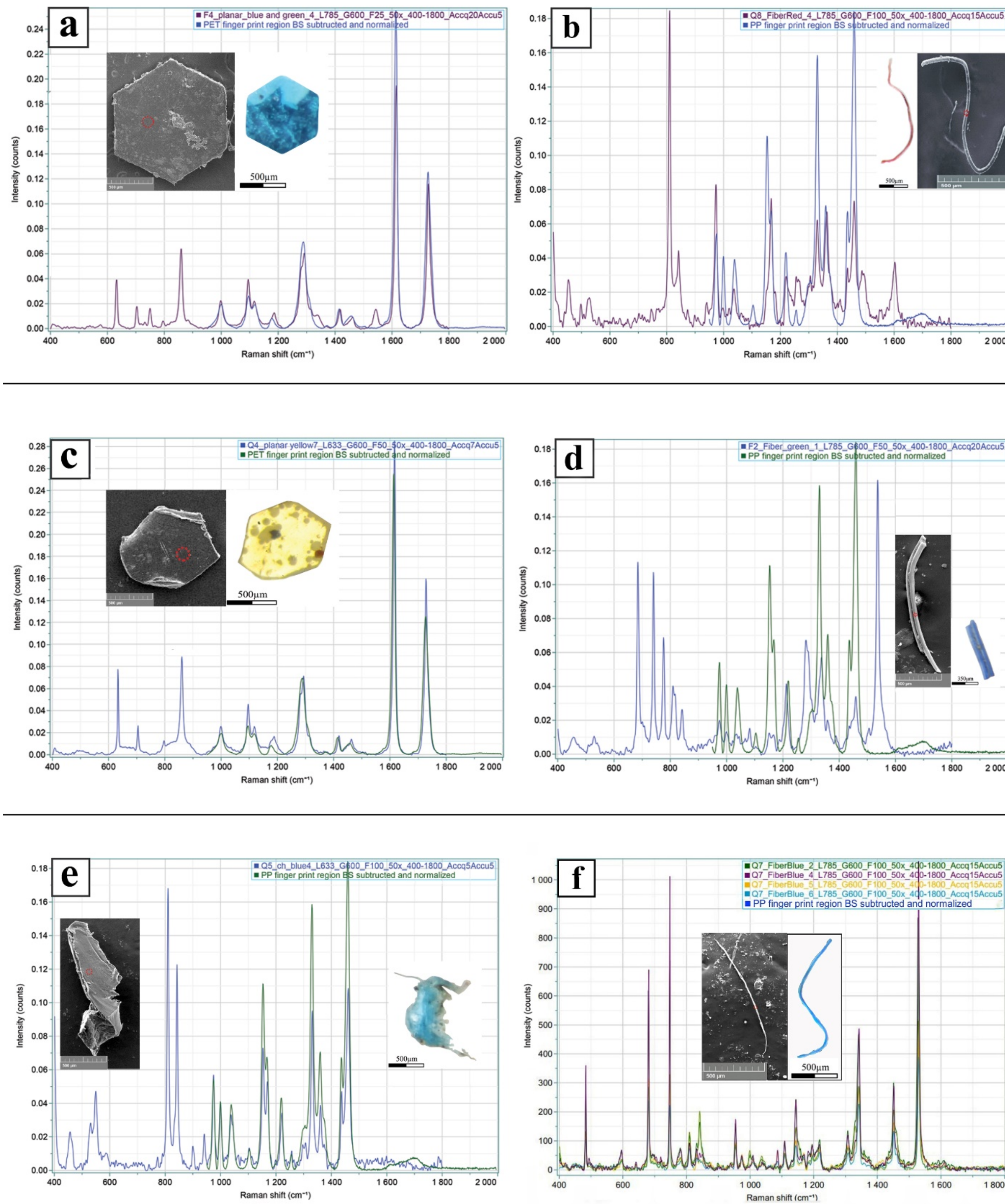

a
b
c
Legend
Zone 1
Zone 2
Zone 3
Sample location
Shape
S i z e
Colour
7 Km
N
29°30'0"N
29°25'0"N
29°20'0"N
52°42'0"E
52°48'30"E
S1
S2
S3
S4
S5
S6
S7
S8
S9
S10
S11
S12
S13
S14
S15
S16
Y(stations)
C1
C2
C3

Figure 7



a
F4_planar_blue and green_4_L785_G600_F25_50x_400-1800_Accq20Accu5
PET finger print region BS subtructed and normalized
500µm
Intensity (counts)
Raman shift (cm⁻¹)
b
Q8_FiberRed_4_L785_G600_F100_50x_400-1800_Accq15Accu5
PP finger print region BS subtructed and normalized
500µm
c
Q4_planar yellow7_L633_G600_F50_50x_400-1800_Accq7Accu5
PET finger print region BS subtructed and normalized
500µm
d
F2_Fiber_green_1_L785_G600_F50_50x_400-1800_Accq20Accu5
PP finger print region BS subtructed and normalized
e
Q5_ch_blue4_L633_G600_F100_50x_400-1800_Accq5Accu5
PP finger print region BS subtructed and normalized
500µm
f
Q7_FiberBlue_2_L785_G600_F100_50x_400-1800_Accq15Accu5
Q7_FiberBlue_4_L785_G600_F100_50x_400-1800_Accq15Accu5
Q7_FiberBlue_5_L785_G600_F100_50x_400-1800_Accq15Accu5
Q7_FiberBlue_6_L785_G600_F100_50x_400-1800_Accq15Accu5
PP finger print region BS subtructed and normalized
500µm

Table 1 Descriptive statistics of MPs concentration. Concentration in Min. Max, Mean, Med and S.D> are given in MPs kg^{-1} solid or l^{-1} liquid in abiotic compartments of the Maharloo Lake. N = number, S.D = standard deviation.

Compartment	N. of MPs	% MPs	Min.	Max.	Mean	Med.	S.D.
All	742	100	10	53	22.5	17	12.9
Surface sediment	245	33.0	11	35	20.4	16	8.9
Lake salt	83	11.2	9	12	10.4	10	0.9
Sludge	155	20.9	50	53	51.7	52	1.5
Lake water	156	21.0	13	30	22.3	24	6.7
Wastewater	103	13.9	34	35	34.3	34	0.6

Table 2 Physical properties and chemical composition of representative MPs in abiotic compartments in the Maharloo lake.

Sample type	Site	Shape	Colour	Polymer type	Elemental composition (mass %)								
					C	O	N	Al	Si	Pb	Cl	Zn	Cu
Sediment	S4	fibre	black	PET	69.2	21.3	4.71	0.7	0.9	-	1.8	1.1	0.2
Sediment	S7	fibre	black	PET	71.3	19.5	5.38	0.3	0.6	-	1.9	0.82	-
Sediment	S14	sheet	white	PP	60.7	22.3	11.27	0.9	1.1	0.3	2.9	0.5	-
Sediment	S16	fragment	blue	PP	64.1	23.6	5.83	0.8	1.1	0.2	2.8	1.14	0.1
Lake water	S4	fibre	red	PS	61.1	21.3	14.69	0.3	0.7	-	1.0	0.93	-
Lake water	S5	fibre	red	PS	62.6	22.4	10.24	0.6	1.8	0.1	1.1	0.97	0.2
Lake water	S15	sheet	yellow	PET	62.6	20.9	9.65	1.0	1.1	0.3	2.9	1.52	-
Lake salt	S8	fragment	white	PS	66.8	23.6	4.73	0.8	1.2	0.2	1.9	0.74	-
Lake salt	S11	fibre	red	PP	74.7	14.9	5.41	0.7	1.5	-	1.5	0.82	0.4
Wastewater	S2	fibre	white	PP	73.2	15.8	5.85	0.9	1.3	-	1.7	1.12	-
Wastewater	S2	fibre	white	PP	72.7	14.7	8.69	0.8	0.9	-	0.9	1.07	-
Wastewater	S3	sheet	blue	PET	59.3	21.8	13.36	0.7	1.2	0.3	1.5	1.33	0.3
Sludge	S1	fibre	black	PET	72.4	17.5	6.74	0.3	0.9	-	0.9	1.22	-
Sludge	S1	fragment	blue	PP	66.2	21.3	6.33	0.6	1.1	0.2	2.9	1.14	0.1
Sludge	S3	fibre	blue	PP	71.6	13.5	9.74	0.8	1.8	0.1	1.3	0.96	0.2

Environmental Toxicology and Chemistry (ET&C)

Microplastics in abiotic compartments of a hypersaline lacustrine ecosystem

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Supplementary Materials/ Data:

Table S1 Concentration (mean/range) and physicochemical characteristics of MPs in abiotic compartments of the Maharloo lake compared with worldwide similar locations. (NI = not-identified)

Location	Sample Type	Concentration	Major shape	Polymer Type	Reference
Maharlu Lake, Iran	Sediment	20.4 ± 9 MPs kg^{-1}	Fibre	PET, PP, PS	Present Study
	Lake water	2 ± 1 MPs l^{-1}	Fibre		
	Lake salt	10 ± 1 MPs kg^{-1}	Fibre		
	Wastewater	3.4 ± 1 MPs l^{-1}	Fibre		
	Sludge	50 ± 2 MPs kg^{-1}	Fibre		
Caspian Sea, Iran	Seawater	710 MPs m^{-3}	Fibre	PET, PS, NY	Nematollahi et al., 2020
	Sediment	15 MPs kg^{-1}	Fibre		
Iran	Lake salt	160 – 980 kg^{-1}	Fragment	PP	Karami et al., 2017
Turkey	Lake salt	8 – 102 MPs kg^{-1}	Fibre	PE, PP	Gündoğdu, 2018
China	Lake salt	43 – 364 MPs kg^{-1}	Fragment, Fibre	CP	Yang et al., 2015
Lake Ontario, Canada	Lake water	0.8 ± 0.7 MPs l^{-1}	Fragment	NI	Grbić et al., 2020
	Wastewater	13.3 ± 15.5 MPs l^{-1}	Fibre		
Taihu Lake, China	Sediment	11.0 – 234.6 MPs kg^{-1}	Fibre	PP, PET, PS	Su et al., 2016
	Lake water	3.4 – 25.8 MPs l^{-1}	Fibre		
Wuliangsuhai Lake, China	Lake water	3.1 – 11.3 MPs l^{-1}	Fibre	PS, PE	Mao et al., 2020
Poyang Lake, China	Lake water	35 – 72 MPs m^{-3}	Fibre	PP, PS, PVS, PE	Jian et al., 2020
	Sediment	41 – 182 MPs kg^{-1}	Fragment		
Veeranam lake, India	Lake water	28 MPs km^{-2}	Fibre	NY, PE	Bharath et al., 2021
	Sediment	309 MPs kg^{-1}			
Bohai Sea, China	Seawater	23 ± 8 MPs l^{-1}	NI	PET, PE,	Feng et al., 2021
	Sea salt	150 ± 28 MPs kg^{-1}	NI		
WWTP in Hong Kong	Wastewater	12.8 ± 5.8 MPs l^{-1}	Fiber	PE, PS	Cao et al., 2020
WWTP in Harbin, China	Wastewater	30.6 ± 7.8 MPs l^{-1}	Fibre	PA, PET	Jiang et al., 2020
	Sludge	46.3 ± 6.2 MPs g^{-1}	Fragment		
WWTP in Madrid, Spain	Wastewater	12.8 ± 6.38 MPs l^{-1}	Fibre	PP, PE, PS	Edo et al., 2020
	Sludge	165 ± 37 MPs g^{-1}	Fibre		