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Microplastic pollution in inshore and offshore surface waters of the southern Caspian Sea

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ABSTRACT

In this study, as the first comprehensive monitoring, the occurrence of microplastics (MPs) in inshore and offshore surface waters of the southern Caspian Sea was investigated. Our data indicated that MPs, which were detected in all the samples, were widely distributed in the thirteen studied stations. Non-normally distribution of the MPs was observed among the studied stations ($p < 0.05$). The average concentration of microplastics in the selected stations was 0.246 ± 0.020 MP/m^3. In most of the transects, negative gradients of MPs from coastal waters to deeper waters were observed. The dominant size and color of MPs in the inshore and offshore water samples was 1000–5000 μm and white-transparent, respectively. Films and fibers constituted about 50% and 40% of the total number of MPs of the water samples, respectively. Also, polyethylene (PE), polypropylene (PP) and polyethylene terephthalate (PET) were the three main polymer types of microplastics in the inshore and offshore surface waters. Our data provide valuable evidence for the comparative assessing of future data regarding decreases or increases of MPs in the southern Caspian Sea.

1. Introduction

Microplastics are plastic litters in the environment with the size <5 mm in diameter (Moore, 2008). Nowadays, marine microplastics pollution is an important environmental issue around the world (UNEP, 2011). Jambeck et al. (2015) assessed that the annual input of MPs to marine environments range between 5 and 13 million tons. The main concern is attributed to their persistence, ubiquity and the potential risk for marine organisms (Kanhai et al., 2018). Microplastics with different sizes have been detected in almost all marine and terrestrial environments even in remote locations such as the Arctic, deep seas, mountain catchments and mountaintop tops (Allen et al., 2019; Napper et al., 2020; Parga Martínez et al., 2020). Microplastics with varying types and density were distributed in all marine habitats including surface water and deep-sea sediments (Buhl-Mortensen and Buhl-Mortensen, 2017). Populated shorelines and beaches, marine gyres and submarine canyons are the main hotspots of marine litters (Brach et al., 2018).

Microplastics have diverse primary or secondary sources and showed different ranges of sizes, shapes and polymer types (Migwi et al., 2020). These plastics may be swallowed by different organisms and transmit through marine food chains from primary producer to human (Mercogliano et al., 2020). Furthermore, by sorption of contaminants from the surrounding environments on their surfaces, MPs can act as a vector for contaminants in the environment and pose health risk to marine biotas and human (Carbery et al., 2018).

Recently, increasing attention has been paid to near-surface layer of the oceans for MPs identification, because the majority of synthetic polymers are concentered in this part of the water column due to density characteristics (Li et al., 2021). The modelling of floating MPs transport on the ocean surfaces indicated that they are more concentrated in the coastal areas and oceanic convergence zones (Zhang et al., 2020). The laboratory and numerical testing of anthropogenic fibers in the Baltic Sea indicated that surface layer of water column have 3–5 times larger fiber abundances than middle layers (Bagaev et al., 2017). Eriksen et al. (2013) estimated that there are more than 2.5×10^5 tons of floated plastic waste on the surface of world oceans. Floating MPs can be vertically transported within the water column through several mechanisms include hydrodynamic factors, biofouling, incorporation into faecal matters and aggregates (Song et al., 2018).

Since the presence and distribution of microplastics in the marine ecosystems have been identified as an important risk to aquatic ecosystems, it is essential to develop monitoring plans to explore and

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identify hotspots of MPs pollution in global aquatic systems.

The south of the Caspian Sea is impacted by intensive tourism activities, densely populated centers, fishing activities and input of polluted effluents from inland basins make it to be candidate for one of the polluted marine environments in the world (Kostianoy and Kosarev, 2005; Ghayebzadeh et al., 2020b; Mehdinia et al., 2020). There are few works which have detected MPs in water, sediments and fishes of Iranian coasts in the Caspian Sea (Ghaffari et al., 2019; Alavian Petroody et al., 2020; Ghayebzadeh et al., 2020a, 2020b; Mataji et al., 2020; Mehdinia et al., 2020; Nematollahi et al., 2020; Rasta et al., 2020; Zakeri et al., 2020; Taghizadeh Rahmat Abadi et al., 2021). In the previously studies, MPs were detected in coastal waters in the restricted areas (Mataji et al., 2020; Nematollahi et al., 2020), whereas their existence, abundance and spatial distribution in offshore zones remains uncertain. All the previously studies in southern Caspian coasts accomplished in the coastal regions of Mazandaran province, whereas, the regions in Guilan and Golestan provinces remain unidentified. The presence of unexplored regions, the lack of comprehensive and baseline MPs data in coastal waters and potential threats posed by MPs to human health and marine organisms confirm the importance of this investigation in the southern Caspian Sea. Therefore, the main scope of this study was to provide an insight into the occurrence, sources, spatial distribution and main features of MPs (including size, color, shape and polymer type) in inshore and offshore surface waters of the southern Caspian Sea.

2. Material and methods

2.1. Study area and field sampling

The Caspian Sea, which is the largest inland water body on the earth, has an area of about 380,000 km^2 and is enclosed by the five countries of Russia, Kazakhstan, Azerbaijan, Iran and Turkmenistan (Kostianoy and Kosarev, 2005; Bastami et al., 2014). It has a salinity of approximately 12 psμ which approximately a third that of mean global seawater (Wikipedia, 2021b). The Caspian Sea is consisted three distinct basins including north, middle, and south (Amirahmadi, 2000; Wikipedia, 2021b). The southern part, which is account for about 66% of the total water volume, is the deepest area of the Caspian Sea, with depths of over 1 Km (Amirahmadi, 2000). The Iranian coastlines of the Caspian Sea stretch approximately 820 km and include the provinces of Golestan, Mazandaran and Guilan (Alizadeh et al., 2018). More than 8 million people live in these provinces (SCI, 2021) and, during New Year and summer holidays, the population is increased by more than 40% (Mehdinia et al., 2020). The main economic activities in the region are agriculture, tourism and fishery (Alizadeh et al., 2018).

In order to study the spatial distribution of microplastics in surface waters of the southern Caspian Sea, 13 inshore and offshore stations along six transects were selected (Table S1). The transects were Lisar (Lis), Kiashahr (Kia), Ramsar (Ram), Chalus-Nowshahr (Cha), Larim (Lar) and Bandare-Torkman (Tor). Seawater pH, dissolved oxygen (DO), salinity and water temperature were determined in situ using Hach multimeter (HQ40d). The geographic coordinates of the studied stations are presented in Table S1. The seawater pH, salinity, DO and water temperature values during the MP sampling were in the ranges of 8.28–8.55, 10.98–12.60 psμ, 10.02–11.30 mg/L and 10.30–15.30 °C, respectively.

To collect MPs floating at the water surface, most researchers use nets such as Neuston (Kor and Mehdinia, 2020), Manta (Gewert et al., 2017; Bakir et al., 2020), plankton net (Castillo et al., 2016; Rasta et al., 2020) and other nets (Ory et al., 2020). The nets can efficiently filter a large mass of water. Other sampling tools such as Niskin (Zhu et al., 2018; Xia et al., 2021) and simple bottle (Nematollahi et al., 2020; Jung et al., 2021) were also reported. The important factor affecting a sampling by net is mesh size of the net (Yutaka et al., 2019). Yutaka et al. (2019) compared two different nets in MPs sampling and stated that, with the same mesh size, there was no statistically significant difference in the responses (the concentration of MPs) obtained by using these two nets (Yutaka et al., 2019). From the viewpoint of harmonizing monitoring methods, using a net with mesh openings of about 300 μm is recommended as it is currently most commonly used method (Eriksen et al., 2018; Yutaka et al., 2019). Net length, boat speed and the weather and sea conditions during the sampling can also affect the MPs sampling and, therefore, should be noted. In this study, a plankton net (length of 170 cm) with a 300 μm mesh size and a circular opening (diameter of 50 cm) which had a sample collector was implemented for water sampling. In order to determine the water volume through the plankton net, a

Table 1
Characteristics of the stations based on color, shape and Raman type of the majority and minority of the observed MPs.

Transect	Station	Major MPs	Type of Major MPs	Other observed MPs	Type of other MPs
Lisar	Lis-1	White fiber	PET	White-transparent film, blue-green cylinder, blue-green fragment and black fiber	White-transparent film (PE) Blue-green fragment (PP)
	Lis-2	White fiber	PET	White-transparent film, black fiber and blue pellet	White-transparent film (PE), Black fiber (PET) and blue pellet (PP)
Kiashahr	Kia-1	White fiber	PET	Black fiber	–
	Kia-2	White fiber and White-transparent film	White fiber (PET) and White-transparent film (PE)	Blue fragment, black fiber, blue fiber, red fiber and red cylinder	Blue fragment (PP and Nylon), blue fiber (PET), red cylinder (PS)
Ramsar	Ram-1	White fiber	PET	Blue fragment and black fiber	Blue fragment (PP)
	Ram-2	White fiber	PET	Red fiber and fragments, White-transparent film	Red fragments (PP) and White-transparent film (PE)
Chalus	Cha-1	White fiber	PET	Black, red and blue fibers, red and blue fragments	Black fiber (PP) and blue fragment (PP)
	Cha-2	Red fiber	PP	White, black and blue fibers	Blue fiber (PP)
Larim	Lar-1	White fiber	PET	Blue and black fibers, blue fragment, White-transparent film	Blue fragment (PP) and White-transparent film (PE)
	Lar-2	White fiber	PET	Blue fragment, red fiber, White-transparent film, yellow fragment	Blue fragment (PP), red fiber (PET), White-transparent film (PE) and yellow fragment (PET)
Bandare-Torkman	GBy	White-transparent film and white fiber	White-transparent film (PE) and White fiber (PET)	Blue film, fiber and fragments, red fiber and cylinder	Blue film (PP)
	Tor-1	White-transparent film, blue fiber and white fiber	White-transparent film (PE), blue and white fibers (PET)	Blue cylinder and fragment, white pellet, red fiber and cylinder, black fiber	Blue cylinder and fragment (PP), white pellet (PE), red cylinder (PS)
	Tor-2	White-transparent film and fragments, blue fragment	White-transparent film and fragments (PE), blue fragment (PP)	White fiber	–

flowmeter was installed across the net opening. The volume of water was computed by multiplying the area of the mouth by the distance of surface water towed. The plankton net was towed at a speed of 2–3 knots. Triplicate samples at each point were collected by the net in sunny days (wind speed of about 3 Km/h and wave height of less than 0.5 m) in February 2019. The mean volume of the filtered waters was approximately 141.37 m^3. It should be noted that the plankton net is not suitable in wavy conditions and in such conditions, other nets such as Neuston should be used. After the towing, the contents of the net were washed with distilled water from the outside of the net into a sample collector (Castillo et al., 2016). The samples were gathered in pre-cleaned 250 mL glass containers and transferred to the laboratory.

2.2. Preparation, observation and identification of microplastics

To remove comb jelly (which is an invasive species in the Caspian Sea), grass, and other large pieces, the water samples were filtered through an approximately 5 mm sieve. Then, each sieved sample was filtered onto ashless S&S filter papers (blue band, grade 589/3, 12.5 mm diameter and <2 μm pore size, Whatman) (Mehdinia et al., 2020). As the impurities in the sieved samples were negligible, chemical pretreatment procedures, which may affect the loss of samples, were not implemented. Besides, the identification of the samples during the visual analysis was not affected by the impurities. Finally, the filters were placed in covered petri dishes and allowed to dry in an oven at 60 °C.

Stereo microscope (HUND) was used to identify the size, color and shape of MPs. In this study, the collected microplastics were sorted according to size (L < 300 μm, 300 μm ≤ L < 500 μm, 500 μm ≤ L < 1000 μm, 1000 μm ≤ L < 3000 μm and 3000 μm ≤ L < 5000 μm), shape (fiber, film, cylinder, pellet and fragments) and color (white/transparent, black/gray, blue/green, red/pink and yellow/orange) (Mehdinia et al., 2020). Also, to verify the observations, polarized light microscopy (PLM) (Olympus, BX41TF) was used. Taking into account the so-called birefringent properties of anisotropic materials such as MPs, PLM was used to confirm the observations (Lusher et al., 2013; Mehdinia et al., 2020). Surface morphological properties as well as major and minor elements of MPs were determined by FE-SEM (TESCAN MIRA3, Czech Republic) equipped with an energy-dispersive X-ray micro analyzer (EDS). Furthermore, micro-Raman spectroscope (Avantes, Netherland) with a laser of 532 nm ± 1 nm and Raman shift of 50–3500 cm^{-1} has been used to identify the polymer types of MPs.

2.3. Statistical analyses

The abundances of microplastic particles were expressed as MPs per cubic meters (m^3) of the inshore and offshore water samples. The graphs were created using Excel 2016 software. The statistical Kolmogorov-Smirnov tests were used to investigate the normality of microplastic data. The SPSS (ver. 26) was used to perform the Kolmogorov-Smirnov test.

2.4. Quality assurance

In order to avoid self and cross-contamination, cotton lab coat, nitrile gloves and glass containers were used throughout the laboratory analysis. The work surface and all equipment were cleaned with ethanol 70%. Aluminum foils were used to cover MP sample containers. To check the possible airborne MP contaminations occurred during the sample preparation and laboratory analysis, three pre-cleaned blank control petri dishes were left uncovered on the lab bench during the experiments. The analysis confirmed no MP contamination in the control petri dishes.

3. Results and discussion

3.1. Microplastic abundances and spatial distribution

Microplastics were detected in all the stations. They were widely distributed in the stations (Fig. 1a). In general, non-normal distribution of the microplastic particles was observed among the stations ($p < 0.05$). The average microplastic concentration in the selected stations was 0.246 ± 0.020 MP/m^3. The abundance of MPs was in the range of 0.056–0.635 MP/m^3. The highest and lowest concentrations of MPs were found at Tor-1 and Kia-1 stations, respectively (Fig. 1a). Tor-1, GBy and Tor-2 were the three stations with higher concentration of microplastics. These stations are situated in the region with higher level of fishing activities. Moreover, inflow of freshwater from rivers such as the Gorganrud, Nokandeh and Qarasu can increase the release of plastics in this transect. The Gorgan bay, which is situated at the southeastern part of the Caspian Sea, is suffered from different environmental stresses such as excess of nutrients, polycyclic aromatic hydrocarbons (PAHs), plastics and heavy metals (Bastami et al., 2012; Araghi et al., 2014; Bagheri et al., 2020). Lis and Lar were the cleanest transects and Tor was the most polluted transect (Fig. 1b). The mean abundance of MPs in the transects followed a descending order: Tor ≫ Kia > Ram ≈ Cha > Lis > Lar (Fig. 1b).

In four of the six transects including Lis, Cha, Lar and Tor, negative gradients of MPs from coastal waters to deeper waters were observed (Fig. 1a and Table S2). The higher concentrations of MPs in the coastal waters of the southern Caspian Sea can be attributed to various sources such as river inputs, untreated sewage inflows, tourism activities, aquacultures and textile industries (Mehdinia et al., 2020). The similar observations were reported in the surface waters of southern Persian Gulf in Qatar (Castillo et al., 2016), Iranian coasts of the Persian Gulf (Kor and Mehdinia, 2020), China (Xia et al., 2021), Atlantic Ocean (Lechthaler et al., 2020), South Korea (Jung et al., 2021), Mediterranean (Pedrotti et al., 2016) and Canada (Desforges et al., 2014). As can be seen from Fig. 1c, the mean concentration of MPs in all the inshore stations was higher than those in the deeper ones. However, more data are required to statistically show this difference.

3.2. Morphological characteristics of MPs

The PLM images of some selected microplastics were shown in Fig. 2. Cross-polarized light (CPL) can affect the structure of the microplastics, and this lead to color changes (Mehdinia et al., 2020). Although, the initial detection of MPs was confirmed using the PLM, it is difficult to identify polymer types using this technique (Underwood et al., 2017).

3.2.1. Size, color and shape of microplastics

The size of MP affects its uptake rate by organisms and consequent adverse biological effects (Lee et al., 2013). By increase in surface area, pharmaceuticals, heavy metals, PAHs and other pollutants can be adsorbed on the hydrophobic surfaces of microplastics (Velzeboer et al., 2014; Pittura et al., 2018; Aliabad et al., 2019; Puckowski et al., 2021). Our data showed that the dominant sizes of collected microplastics in the inshore and offshore water samples was 1000–5000 μm (accounting for about 60% of the total number of MPs) (Fig. 3a and Table S.3). Microplastics larger than 1000 μm were dominant in most of the stations. The variety of sizes in the eastern transect (Tor) and western transect (Lis) was more than the others.

The color of microplastics may be changed in surface waters by processes such as bleaching and weathering (Stolte et al., 2015). In this study, the dominant color of microplastics in the inshore and offshore water samples was white-transparent (accounting for about 77% of the total number of MPs) (Fig. 3b and Table S.4). The dominance of white-transparent microplastics has been reported in coastal waters of the southern Caspian Sea (Mazandaran providence) (Mehdinia et al., 2020; Nematollahi et al., 2020). As can be seen from Fig. 3b, the

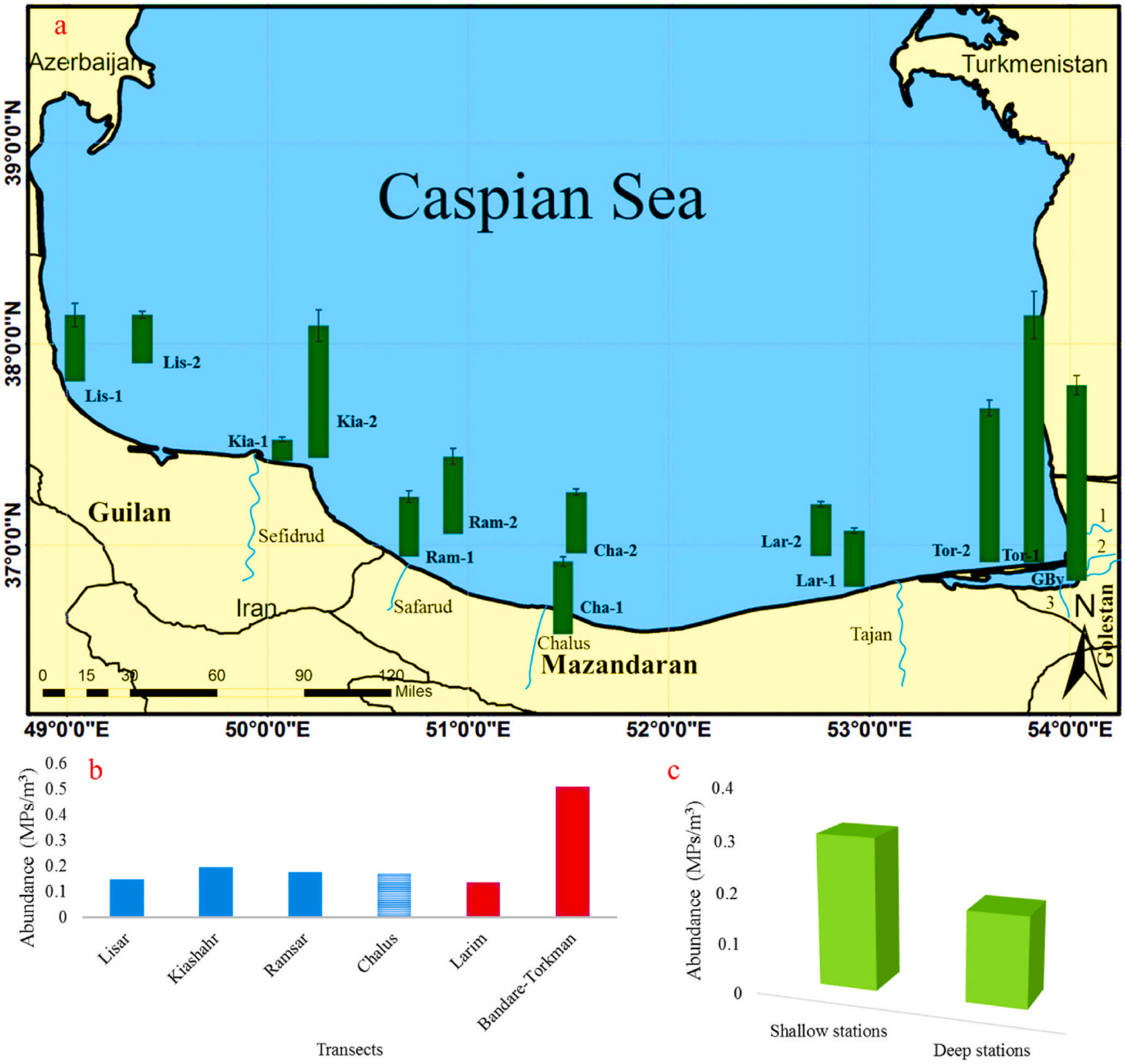

Fig. 1. (a) Spatial distributions of MPs collected from surface waters of the southern Caspian Sea. 1, 2 and 3 in Tor transect are Gorganrud, Qarasu and Nokandeh rivers, respectively; (b) Mean abundance of MPs in each transect; (c) Comparison of the mean abundance of all shallowest stations with the mean abundances of all deepest stations.

Table 2
Comparison of the abundances and major shapes, colors and types of observed MPs in this study with some other marine research around the world.

Location	Location specification	Abundance (MPs/m^3)	Major shape	Dominant color	MPs dominant type	Reference
Iran	Inshore and offshore waters of the southern Caspian Sea (including Guilan, Mazandaran, and Golestan provinces)	0.246	Film and fiber	White/transparent and blue	PE, PP and PET	Current study
Iran	Anzali Wetland	1.25	Fiber	Red, black and blue	PP, PE and polyester	Rasta et al. (2020)
Iran	Southern Caspian Sea coastal waters (Mazandaran province)	–	Fragments and foams	White and milky	PE, PP and PS	Mataji et al. (2020)
Iran	Southern Caspian Sea coastal waters (Mazandaran province)	–	Fiber	Black	PET, PS and nylon	Nematollahi et al. (2020)
Iran	Chabahar Bay, Gulf of Oman, nearshore waters	0.49	Fiber	White, blue and red	PE and PP	Aliabad et al. (2019)
Baltic Sea	Kiel Fjord	0.04	Fragment	Green	HDPE and PP	Ory et al. (2020)
Qatar	Qatar's Exclusive Economic Zone	0.71	Granular and fibrous	Blue and opaque white	PP	Castillo et al. (2016)
Atlantic Ocean (Portugal)	Atlantic Ocean south of Algarve Coast	1.36	Fiber	Black, blue and pink	HDPE and PP	Lechthaler et al. (2020)
Black Sea	Black Sea	0.6×10^3	Fiber and film	–	–	Aytan et al. (2016)

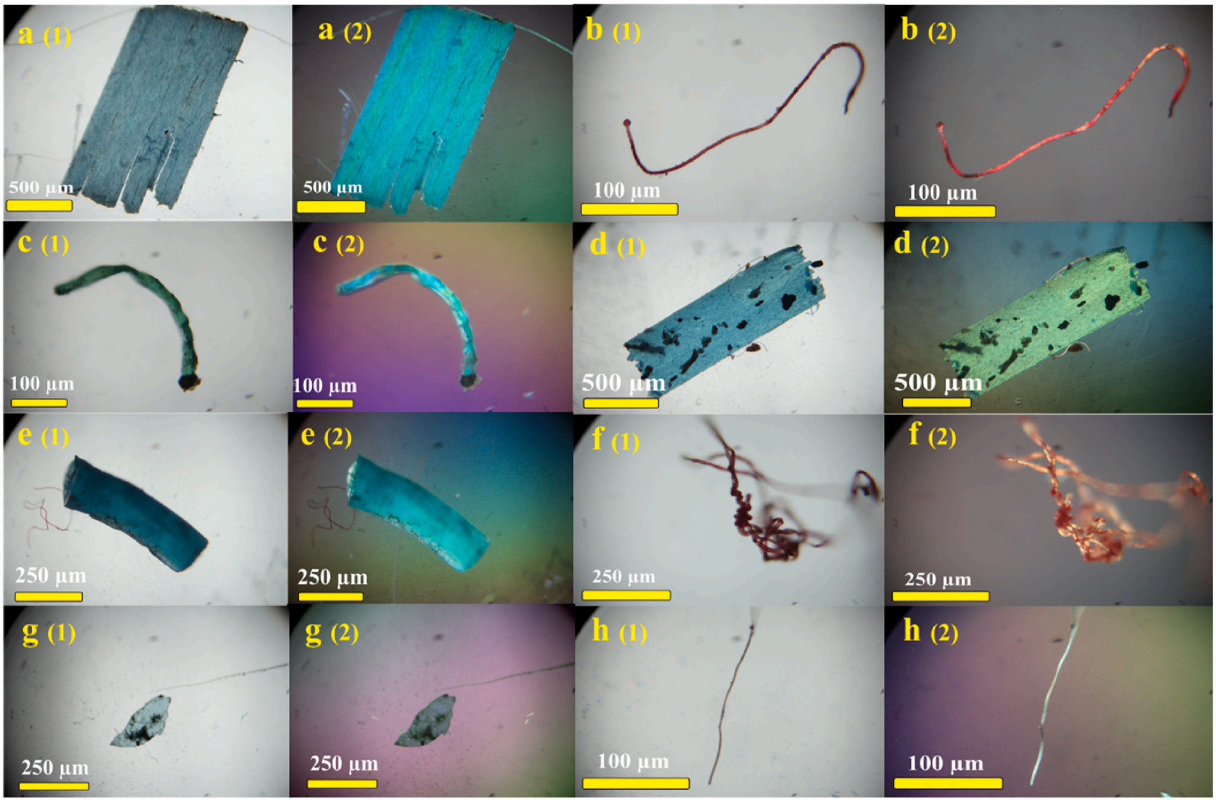

Fig. 2. PLM images of typical MPs collected from the surface waters. In each section, (1) and (2) refer to Plane-polarized light (PPL) and Cross-polarized light (CPL) images, respectively.

white-transparent color MPs were dominant at all stations except Cha-2 (which red-pink was prevailing). The yellow/orange color MPs were only detected at Lar-2 stations. Also, the variety of colors in the stations of Kia-2, Cha-1, Cha-2 and Lar-2 was more than the others.

Floating and transportation behaviors of microplastics can be influenced by shape (Kowalski et al., 2016). In this study, films and fibers constituted about 50% and 40% of the total number of MPs of the water samples, respectively (Fig. 3c and Table S.5). Several researchers have been reported the prevalence of fibers and films in coastal seawater, fish tissues and coastal sediment of the southern Caspian Sea (Ghayebzadeh et al., 2020a; Mataji et al., 2020; Mehdinia et al., 2020; Nematollahi et al., 2020; Zakeri et al., 2020). Fibers, fragments, cylinders, pellets and films were observed in the inshore and offshore surface waters. Fibers and films were dominant shapes at all the stations (Fig. 3c). The majority of the MPs found in the Tor transect were in the form of films. The cylinder form of MPs was only detected at Lis-1, Lar-1 and Tor-1 stations. The pellets were only detected at Lis-2 and Tor-1 stations. The shape of microplastics can fairly provide information on their origin (Shim et al., 2018). Fabrics, ropes, nets, and filaments used in aquaculture farms and fishery are the main sources of fibers (Jang et al., 2014; Shim et al., 2018). The high abundance of fibers in this study may come from fishing activities and municipal sewage. fragmentation is thought to be the main source of fragments (Shim et al., 2018). Films generally originate from packaging material and plastic bags (Shim et al., 2018). Pellets can be considered as primary plastics (Shim et al., 2018). High proportions of fragments, films and fibers in marine environments specify that secondary MPs contribute to MP concentration more so than do primary MPs (Browne et al., 2011; Shim et al., 2018).

The contribution of each shape of microplastics in different sizes of inshore and offshore water samples was also investigated (Fig. S1). The majority of MPs with sizes less than 500 µm were fragments and films, whereas the highest percentage of fibers was observed in sizes of L $\geq$ 1000 µm. The most varied shapes of MPs were observed in sizes of 300–500 µm. Also, films were highest in the middle size fraction of the inshore and offshore MPs (500–1000 µm).

As the appearance of plastic and prey is similar, colors cause organisms to misidentify microplastics as food (Aliabad et al., 2019; Kor and Mehdinia, 2020). In this study, blue-green, white-transparent and red-pink colors were more detected in fragments, films and fibers, respectively (Table S.6). Also, black-gray and yellow-orange colors were only observed in fibers and fragments, respectively. Anthropogenic sources such as artificial fabrics and also fishing activities could be the main reason for high concentration of colored fibers in marine environments (Rasta et al., 2020).

3.2.2. *Raman micro-spectroscopy*

Compared with FTIR spectroscopy, Raman micro spectroscopy shows higher sensitivity, narrower spectral bands, better spatial resolution, lower water interference and wider spectral coverage (Ivleva et al., 2017; Araujo et al., 2018). The Raman spectra of some microplastics which identified in the inshore and offshore surface waters were shown in Fig. 4(a–c). PE (40%), PP (~31%) and PET (~22%) were the three main polymer types of microplastics in the inshore and offshore surface waters (Fig. 4d). The major types of MPs in the inshore and offshore surface waters were shown in Table 1. In general, the MP type followed a descending order: white-transparent films (PE) > white fibers (PET) > blue-green fragments (mostly were PP) > blue fibers (PET and PP) > red fibers (PET and PP) > black fibers (PET and PP) > white pellet (PE), red cylinder (polystyrene (PS)), red fragment (PP), yellow fragment and blue pellet (PP). Some polymer types provide information regarding their source (Shim et al., 2018). For instance, PE is primarily used for packaging (plastic films, plastic bags and bottles) (Geyer et al., 2017). PP is released from municipal sewage or from ropes and nets used in boats and ships (Mehdinia et al., 2020). Plastic bottles and artificial fibers are the main consumers of the PET (or polyester) polymer in the world (Ji, 2013; Wikipedia, 2021a).

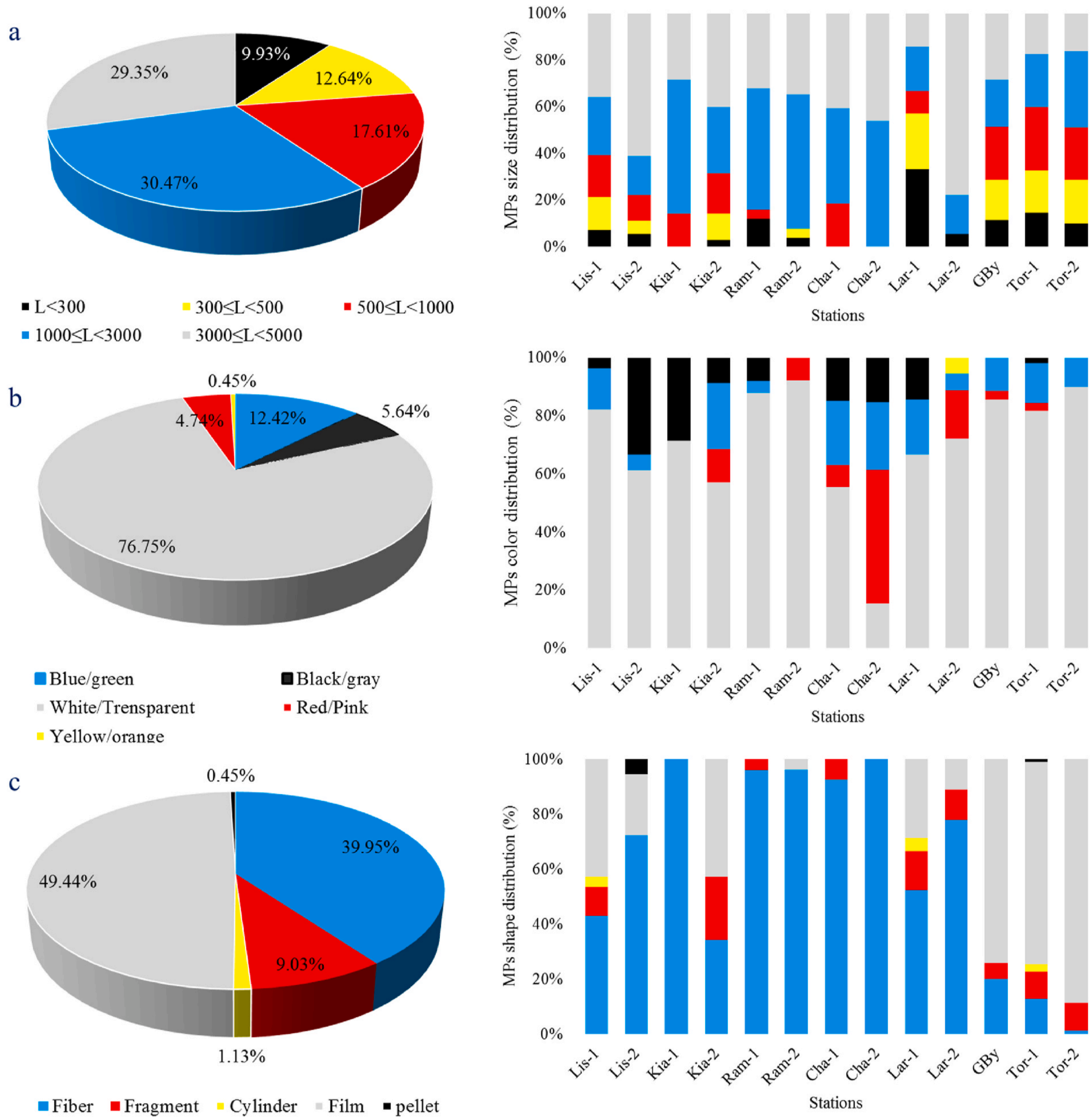

Fig. 3. Proportion (%) of the observed sizes (a), colors (b), and shapes (c) in the total collected MPs. Size, color and shape distributions of MPs are shown in the right sides of (a), (b) and (c) sections, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Several studies were used Raman micro-spectroscopy or FT-IR spectroscopy to identify polymer types of MPs in the coastal waters and sediments of the southern Caspian Sea (Ghayebzadeh et al., 2020a; Mataji et al., 2020; Mehdinia et al., 2020; Nematollahi et al., 2020). Raman micro spectroscopy is an indispensable tool to study very small MPs, where FT-IR technique is entirely insufficient (Araujo et al., 2018). However, regardless of the techniques used for detection, PE, PP, PET and PS were the main polymer types of MPs of the surface waters and sediments of the southern Caspian Sea (Table S.7).

3.2.3. SEM-EDS analysis

The SEM images of some selected MPs from the inshore and offshore water samples were shown in Fig. 5. Cracked, rough, edged and smooth structures were visible. Long-term oxidation, polymer aging and mechanical breakage of larger plastics in the marine environments are the signs of these structures (Abbasi et al., 2018; Mehdinia et al., 2020). The structures increase microplastic's capability to adsorb various contaminants such as heavy metals, antibiotics and PAHs (Abbasi et al., 2017, 2019; Magadini et al., 2020; Mehdinia et al., 2020; Puckowski et al., 2021).

The EDS analysis specified that carbon (C) and oxygen (O) were the major microplastic elements of the inshore and offshore water samples. The EDS analysis showed that all fragments and films had a strong C peak (70–88%) followed by a low O peak (12–28%). Also, the EDS analysis indicated that, in the fibers and cylinders, carbon (47–70%) and oxygen (29–51%) were the main peaks. Presence of high level of oxygen in microplastics may a sign of degradation. Canopoli et al. (2020) indicated that, in the plastic surface alteration, O was the major element. They showed that the carbonyl index (CI) of plastic samples buried for less than 10 years and fresh materials was between 1.5 and 2 times lower than the CI of plastic samples buried for more than 10 years. Low peaks of Ca, K, Fe, Si, Ti and Na were also observed in some samples, which

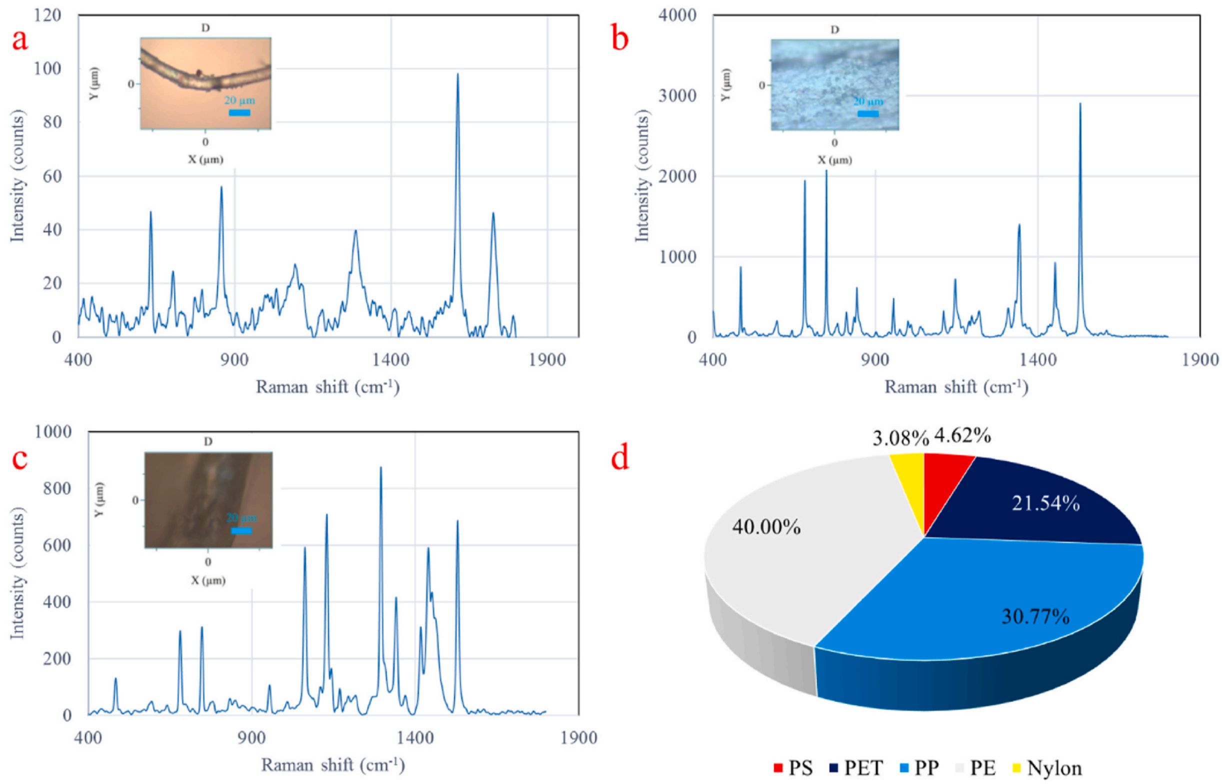

Fig. 4. (a–c) The micro Raman spectra of representative PET (Fiber), PP (Fragment) and PE (Film) MPs, respectively. (d) Proportions (%) of the composition of MPs found in the study area.

may indicate the presence of additives or adsorption from external environments (Ivar do Sul and Costa, 2014; Abbasi et al., 2019).

3.3. The sources of MPs

The United Nations Environment Programme (UNEP) estimated that the main potential sources of MPs in Caspian Sea are coastal tourism, fishing activities, input from rivers, urban solid wastes, shipping and oil/gas exploitation (UNEP, 2009). The contribution of these sources were variable among the different sampling transects (Table S1). The higher concentration of MPs in the Tor transect could be due to the prevalence of fishing (in all months of the year) and tourism activities in this area. Moreover, the existence of permanent rivers, including Gorganrud, Nokandeh and Qarasu, can contribute to the entry of more MPs to this transect. Almost all of the cities located along the southern Caspian Sea coastline have poor policies for the management of coastal and municipal wastes so that vast amounts of MPs can find their way to the sea environment (Ghaffari et al., 2019). Furthermore, hydrodynamics which is reflected in general anti-clockwise circulation of water, can transfer MPs from western parts to eastern parts of the southern Caspian Sea (Mehdinia et al., 2020). The predominant color of MPs in this transect and other transects (except Cha-1 station) was white (Fig. 3b). However, exposure of MPs to sunlight causes photobleaching of their color (Firdaus et al., 2020; Nematollahi et al., 2020). Therefore, caution should be taken when interpreting the color results. The major shape of MPs in the Tor transect was film, which proves that probably tourism activities have a large impact on MP pollution in this region (Mohamed Nor and Obbard, 2014). Interestingly, the abundance of fiber MPs in Tor transect increased in the order of GBy > Tor-1>Tor-2. Fishing activities, especially near the mouth of Gorgan bay, and river inflows can be the sources of fiber MPs in this area.

Fishing activity (from early October to early April) can be the source of MPs in the Lar transect. Also, there was a marine fish cage culture between Lar-1 and Lar-2 stations which can contribute to MP contamination.

The central part of south Caspian coast including Ram and Cha regions is the most visited area in Iran (Mehdinia et al., 2020). Moreover, extensive fishing activities can be the other source for MPs in this area. The major shape of MPs in the Ram and Cha transects was fiber. Also, film (in Ram-2 station) and fragment (in Ram-1 and Cha-1 stations) shapes of MPs were detected. These observations support the hypothesis that fishing and tourism activities as well as the inflows of Safarud and Chalus rivers have large impacts on MP pollution in these regions. Furthermore, there were marine fish cage cultures close to Ram-2 station and between Cha-1 and Cha-2 stations, respectively.

Fishing and tourism activities as well as the inflow of Sefidrud river can play important roles in increasing MPs in Kia transect. The high abundance of MPs in Kia-2 station can be attributed to several parameters including Sefidrud river inflow, general westward current of surface water in the southern Caspian coasts, water bottom topography and coastlines morphodynamic (Mehdinia et al., 2020). Sefidrud is the second longest river in Iran and the major river in the south Caspian coast (Alizadeh et al., 2008). Furthermore, there was marine fish cage culture near Kia-2 station which can contribute to MP contamination.

The western coasts of the southern Caspian Sea, including Lis area, are less developed regions with lower tourism activities than the other sampling sites. However, fishing activity is the main source of MPs in this transect. Also, this station is the closest sample location to input of the general current of surface water from Azerbaijan (and especially from Baku) to the southern coasts of Caspian Sea.

Finally, it should be noted that there is no specific (concentrated) fishing port in the southern coasts of the Caspian Sea. Furthermore, the main commercial ports in this region are Anzali (near Kia transect), Nowshahr (near Cha transect) and Amirabad (near Lar transect). There is no data available on the impacts of these ports on MP abundances.

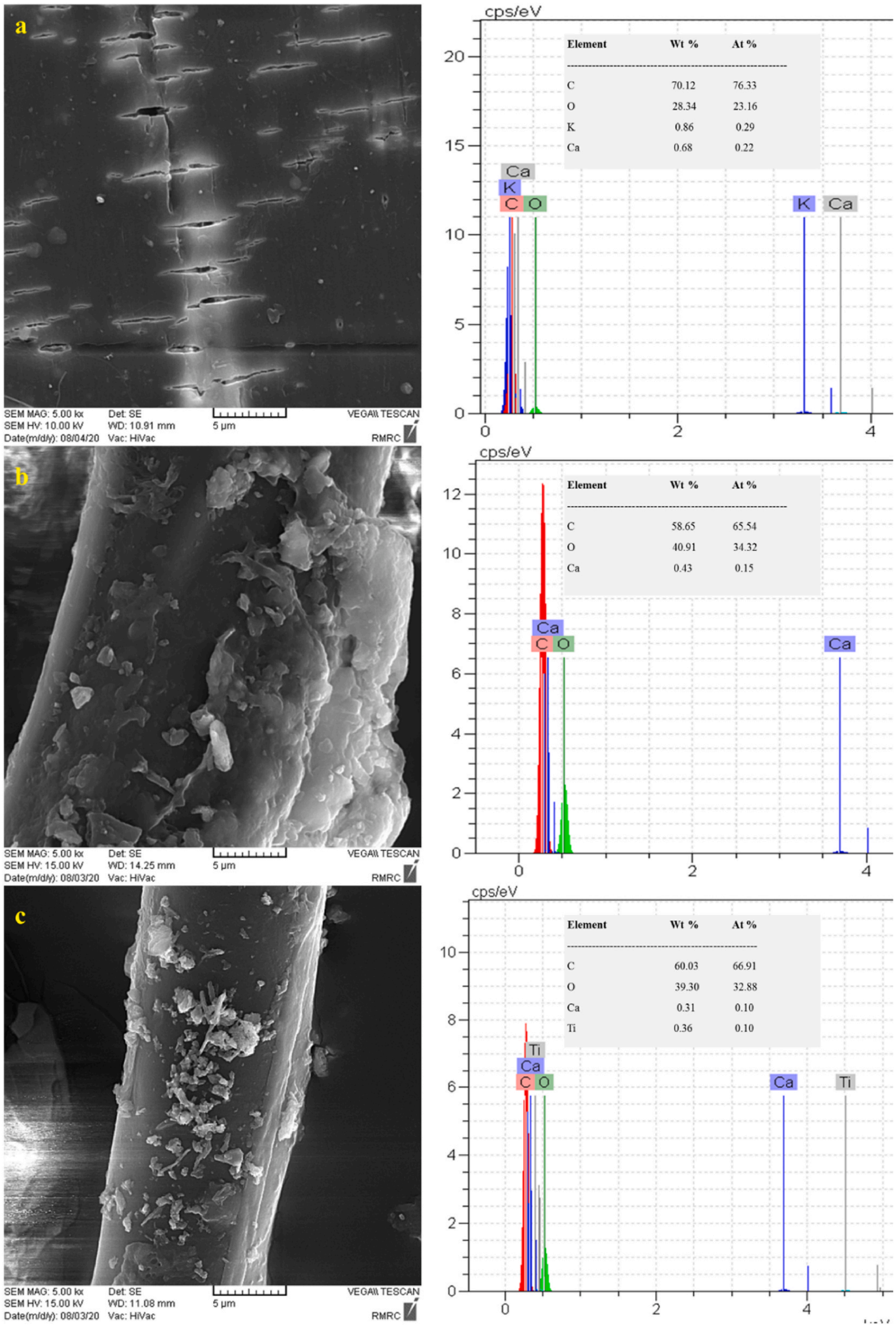

Fig. 5. SEM/EDS analyses of representative MPs found in the surface waters of the southern Caspian Sea: (a) blue fragment, (b) white fiber and (c) red fiber. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.4. Status of microplastics in surface waters of the southern Caspian Sea relative to other regions

Nets with different mesh sizes and niskin bottles were used for MP samplings in surface waters (Aliabad et al., 2019; Kor and Mehdinia, 2020; Mataji et al., 2020; Nematollahi et al., 2020; Rasta et al., 2020). Thus, it is not easy to compare MP concentrations among different studies because of the lack of standardized procedures for sampling (Shim et al., 2018).

As can be seen from Table 2, MP contamination level in the surface waters of the southern Caspian Sea (0.246 MP/m^3) was higher than those of Baltic Sea (Ory et al., 2020). The abundance of MPs in this study was of the same order of magnitude as the MP abundance in the Persian Gulf (Castillo et al., 2016), Atlantic Ocean (Lechthaler et al., 2020), Gulf of Oman (Aliabad et al., 2019) and Anzali Wetland (Iran) (Rasta et al., 2020) but far lower than the MP abundance in Black Sea (Aytan et al., 2016). There is a limited information on microplastic contaminations in surface waters of the Caspian Sea (Mataji et al., 2020; Nematollahi et al., 2020). It should be noted that, both the previously studies were performed in the nearshore and inshore seawater. Mataji et al. (2020) used Neuston net for MP sampling from surface waters. However, Nematollahi et al. (2020) used bottles for water samplings. Furthermore, the previously studies performed in the Mazandaran province, whereas our samplings were performed in entire southern part of the Caspian Sea including Golestan, Mazandaran and Guilan provinces. As can be seen from Table 2, in most of the previously studies, fiber was the dominant shape of MPs. Also, polyethylene and polypropylene were the most reported types of MPs not only in the surface waters of the southern Caspian Sea but also in other marine regions of the world (See Table 2).

4. Conclusion

The Caspian Sea, which is a closed water body, is polluted by different contaminations such as MPs. Our data provide baseline information of MPs in surface waters on a large geographic area of the southern Caspian Sea. MPs were identified in all samples and widely distributed in the stations. The abundance of MPs in this study was of the same order of magnitude as the MP abundance in the Persian Gulf (Castillo et al., 2016), Atlantic Ocean (Lechthaler et al., 2020), Gulf of Oman (Aliabad et al., 2019) and Anzali Wetland (Iran) (Rasta et al., 2020) but far lower than the MP abundance in Black Sea (Aytan et al., 2016). The dominant shapes, color and polymer types of MPs were films and fibers, white-transparent and PE and PP, respectively. We showed that in addition to urbanization, the distribution of MPs in the southern Caspian Sea may be influenced by fishing, tourism activity and untreated rivers. The regions with higher contribution of anthropogenic activities such as Gorgan bay and Ramsar were hot spots of MPs pollution. Therefore, it is necessary to take preventive action to reduce or at least maintain current MP pollution levels to protect such environments in the future.

Our data provided valuable evidence for the comparative analysis of future data regarding decreases or increases of MPs in the surface waters of the southern Caspian Sea. To facilitate our understanding of source identification of MPs, and to identify the potential for MP associated pollutant transfer in marine food chain, this investigation on the prevalence of MPs in the southern Caspian Sea basin will be extended to include marine sediments and biota in future studies.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2021.130896.

CRediT author statement

Ahmad Manbohi: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Project administration; Software; Supervision; Validation; Visualization; Writing – original draft., Ali Mehdinia: Conceptualization; Resources; Methodology. Reza Rahnama: Formal analysis; Data curation; Visualization. Reza Dehbandi: Writing – original draft, Formal analysis.

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