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Prevalence of microplastics in the marine waters of Qatar

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ABSTRACT

Microplastics are firmly recognized as a ubiquitous and growing threat to marine biota and their associated marine habitats worldwide. The evidence of the prevalence of microplastics was documented for the first time in the marine waters of Qatar's Exclusive Economic Zone (EEZ). An optimized and validated protocol was developed for the extraction of microplastics from plankton-rich seawater samples without loss of microplastic debris present and characterized using Attenuated Total Reflectance-Fourier Transform Infrared spectroscopy. In total 30 microplastic polymers have been identified with an average concentration of 0.71 particles m^{-3} (range 0–3 particles m^{-3}). Polypropylene, low density polyethylene, polyethylene, polystyrene, polyamide, polymethyl methacrylate, cellophane, and acrylonitrile butadiene styrene polymers were characterized with majority of the microplastics either granular shape, sizes ranging from 125 μm to 1.82 mm or fibrous with sizes from 150 μm to 15.98 mm. The microplastics are evident in areas where nearby anthropogenic activities, including oil-rig installations and shipping operations are present.

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1. Introduction

Worldwide plastic production is currently about 300 million tons per annum, with global plastic consumption increasing by about 4% per year (Plastics Europe, 2014/2015). Plastic is considered as the primary material for the 21st century due to its widespread application in industrial and consumer products. However, when discarded inappropriately, it becomes litter with persistent and multi-faceted environmental impacts.

The issue of plastic pollution in the marine environment is of increasing global concern. About 62% of plastic waste generated (out of a 25.2 Mtonne annually, average estimate from 2006 to 2012) disposed of annually, is recovered for recycling and energy recovery processes, but the remainder is improperly disposed of into the environment (PlasticsEurope, 2014/2015). Plastic debris reaches the ocean from inland urban areas via surface drainage systems, as well as directly from anthropogenic activities such as: fishing; discharge from shipping; and the intentional and accidental release of domestic, agricultural, and industrial sewage and wastewater effluent (Horsman, 1982; Galgani et al., 2000; Ng and Obbard, 2006; Andrady, 2011). Wind patterns, ocean hydrodynamics, and sea floor morphology are all important vectors that influence the dispersion of plastic debris in the marine environment (Wright et al., 2013; Isobe et al., 2014; Ioakeimidis et al., 2014; Tubau et al., 2015).

The environmental transport of plastic debris is greatly affected by particle size, shape, density and polymer type (Wright et al., 2013; Isobe et al., 2014). Discarded plastic is now prevalent in the ocean as: floating litter on the ocean surface (Barnes and Milner, 2005; Barnes et al., 2009); stranded litter on coastal shorelines (Barnes and Milner, 2005; Thiel et al., 2013); and as sunken litter on sea floor bottom sediments (Galgani et al., 2000; Schulz et al., 2015). Plastic polymers that are positively buoyant in seawater (sp. gr. of seawater is ~ 1.025) are retained at the sea surface, then become dispersed on the water surface (pelagic) before entrapment in areas of low circulation, and then finally sink after further entanglement and biofouling (benthic) (Galgani et al., 2000; Morét-Ferguson et al., 2010; Ioakeimidis et al., 2014). Denser polymers such as nylon (sp. gr. ~ 1.29), polyethylene terephthalate (sp. gr. ~ 1.37), and polyvinyl chloride (sp. gr. ~ 1.38) tend to submerge in the water column, and can reach the marine sediment (Andrady, 2011; Claessens et al., 2013). As plastics typically have an intrinsic durability, water insolubility and slow degradation rates, debris has now become both ubiquitous and persistent in the marine environment. Over time, larger plastic pieces disintegrate into smaller fragments via: photolytic UV radiation; oxidation; hydrolysis; mechanical forces; as well as thermal and biological degradation processes that ultimately result in the generation of microplastics (Browne et al., 2007; Barnes et al., 2009; Andrady, 2011). Several studies have reported a variety of microplastic particle sizes ranging from <10 mm (Graham and Thompson, 2009), 2–6 mm (Derraik, 2002), <2 mm (Browne et al., 2011) and <1 mm (Claessens et al., 2011), <500 μm (Browne et al., 2010), <2 μm (Frias et al., 2010; Browne et al., 2010). Microplastics, as defined by the National Oceanic and Atmospheric Administration (NOAA), comprise of plastic particles that are <5 mm in size.

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Microplastics pose a more serious threat to marine biota than larger plastic debris as they are often in the same size-range as natural food items, thus leading to their ingestion when mistaken as food (Boerger et al., 2010; Lusher et al., 2013; Bond et al., 2013). As a result, marine organisms are particularly susceptible to microplastic ingestion with indirect consequent effects on organisms at higher trophic levels via bioaccumulation. Furthermore, due to their hydrophobic nature, microplastics have been found to concentrate a wide-range of organic contaminants such as: PCBs, PBDEs, PAHs, DDT, and BPA (Hirai et al., 2011; Bakir et al., 2014), trace metals (Aston et al., 2010; Holmes et al., 2012); as well as additive-derived chemicals (e.g. phthalates, nonylphenol) (Teuten et al., 2007; Mathalon and Hill, 2014). Due to ingestion, microplastics therefore serve as a vector for the transfer of contaminants into marine biota. This has been reported to result in disruption of the endocrine system, toxicological hazards, and the bioaccumulation of organic pollutants in the marine food chain (Teuten et al., 2007; Hirai et al., 2011; Gassel et al., 2013).

Microplastic abundance in the marine environment has been shown to have a positive correlation with human population density in the adjacent coastal catchment. Increased human population in coastal areas generally leads to an increase in plastic litter generation, and consequent prevalence of microplastics in coastal waters (Depledge et al., 2013).

In the Middle East there is very limited reported data on microplastic pollution, although the region accounts for about 7.3% of global plastic materials production (PlasticsEurope, 2014/2015). Increased population levels, coupled with strong economic development, is expected to coincide with increased litter generation, including plastic. Qatar is a low-lying peninsula nation situated midway along the western coast of the semi-enclosed Arabian Gulf. Its marine environment is particularly susceptible to marine debris due to the country's increased modernization and economic development. Qatar's marine Exclusive Economic zone (EEZ) extends to approximately 32,000 km² (15% of the Gulf), and includes a 563 km long coastline (Jones et al., 2002; UNCSD, 1997). Although the country's marine ecosystem is a valuable resource for fisheries, tourism and recreation it co-exists with a growing

human population and an expansion of coastal petrochemical industries including oil and gas-rig operations, harbors, port facilities, and land reclamation. Environmental impacts from these anthropogenic activities include: discharge from coastal dredging operations; effluents from power and desalination plants, petroleum industries, and domestic sewage; increased shipping activity, as well as indiscriminate on-shore and offshore litter disposal. Moreover, Qatar's marine hydrologic cycle is influenced by extreme high temperatures during the summer season, when atmospheric temperature increases to an average daily maximum of 46 °C, and sea temperatures reach up to 35 °C. In contrast, during the winter season, the average daily maximum lowers to 10 °C and sea temperature reduces to 15 °C. High evaporation rates occur during the summer causing water salinity to rise to 39–41 ppt, while the prevailing northwesterly Shamal winds during the winter months cause sea turbulence which affects sea currents, generating waves of up to 5 m (Jones et al., 2002; UNCSD, 1997). These hydrodynamic conditions are likely to affect the quantity and size distribution of plastic debris.

Knowledge of the extent and impact of microplastic pollution in Qatar's marine environment is still largely speculative, where no baseline data currently exists for Qatar's EEZ. This report is the first documented evidence for the presence of microplastics in the marine waters of Qatar's EEZ. The study also led to the development of an optimized protocol for the extraction of microplastics from plankton-rich seawater.

2. Materials and methods

2.1. Field sampling

To investigate the presence and distribution of microplastics, marine sampling was conducted using the research vessel of Qatar University, the RV Janan. Surface water samples were collected from 12 marine stations within the northeastern section of Qatar's EEZ in May 2015 (see Fig. 1). All samples were collected using a plankton tow-net (Ivar do Sul et al., 2013; Hidalgo-Ruz et al., 2012) attached to the research vessel.

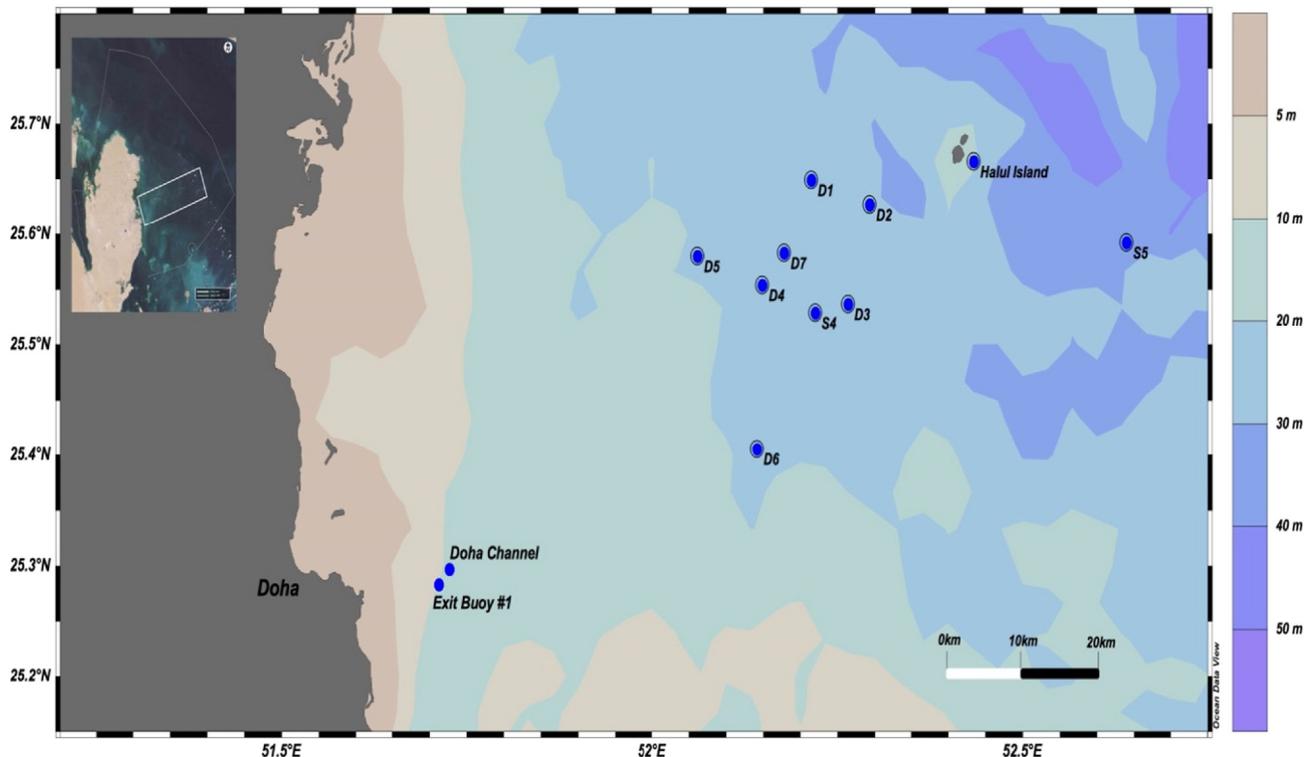


Fig. 1. Geographical location of sampling stations.

The net has a conical-cylindrical design with a 0.5 m diameter mouth, a 2 m length, and a mesh size and cod end of 120 μm . The mesh size of the net controls the lower limit of the micro-particles collected (Isobe et al., 2014). The net was submerged 0.25 m beneath the sea surface, and towed for approximately 5–7 min at a speed of 1–2 knots. A flow meter (Sea-Gear MF315) was fitted to the mouth of the net, and used to measure the volume of water passing through the net during sampling. After sample collection, the net was washed with pre-filtered distilled water (0.45 μm Millipore membrane filter), and its content transferred to a 250 mL glass container which was refrigerated (4 °C) and returned to an onshore laboratory at Qatar University for microplastic examination.

2.2. Digestion and method validation

As the collected samples were rich in plankton, visual inspection using the microscope could not be the only method relied upon for isolating microplastics (Fig. 2). Plankton present in seawater samples may readily mask the identification of microplastics (Cole et al., 2014). Especially if the color of the particle matches that of the biological material. Moreover, any particle that has ambiguous characteristics renders it more challenging to identify accurately. To avoid an underestimation of the quantity of microplastic particles present, it was therefore necessary to perform a digestion of the naturally-occurring biological materials in the sample. Several digestion protocols have been reported in previous studies (Avio et al., 2015; Nuelle et al., 2014; Cole et al., 2014; Claessens et al., 2013; Desforges et al., 2014). As this was the primary investigation of microplastics in Qatar's marine waters, an improved and validated protocol was developed and optimized for the extraction of microplastics from the plankton-rich seawater samples in the Qatar EEZ. The protocol involved sample centrifugation, chemical digestion followed by filtration, microscopic examination, and finally FTIR analysis.

The development of a digestion protocol was conducted using solutions of 1 M NaOH, 10 M NaOH, and 16 M HNO₃. Twenty milliliter of each solution was spiked with known quantities of reference polymer pellets, of varying diameters (63 μm to 4.70 mm), as well as some commercially available plastic polymers i.e. polyethylene, polyethylene terephthalate, polystyrene cut into 1–5 mm sizes. The reference polymer pellets used were manufactured by Sigma Aldrich, BDH Chemicals, Sabic, and Qatar Petrochemical Company and consisted of different sized polymers, including: polyvinyl chloride (63–125 μm); polyvinyl alcohol (125 μm –2 mm); polytetrafluoroethylene (250 μm –1 mm); nylon (2.30 mm); polystyrene (3.19 mm); low density polyethylene (3.97 mm); and low-low density polyethylene (4.70 mm). The protocol consisted of digestion at room temperature for 24 h, followed by heating

at 60 °C for 2 h, and then further heating at 100 °C for 1 h (Cole et al., 2014; Claessens et al., 2013). The solution producing the most efficient digestion (expressed as percentage particle recovery) was used to digest the plankton-rich sample. Differences in particle recovery efficiency were verified using ANOVA with Tukey HSD post-hoc analysis. The homogenized sample plankton biomass was then subjected to the most efficient digestion treatment method, where 20 mL of the digestion solution per 0.1 g dry weight of plankton sample was digested, as per the optimized protocol.

For quality control purposes and avoid air contamination, the analysis was performed inside the fumehood i.e. preparation, digestion and filtration. A 11 μm Whatman filter paper in a petri dish was used as a control blank (negative control) and placed inside the fumehood and on the work station. Work surfaces were cleaned with alcohol, and nitrile gloves were worn at all times. All materials used for sampling, extraction and analysis were made of non-plastic material, except for the plankton net tow, which was examined thoroughly to ensure that no loose plastic fragments were present. All materials used were rigorously cleaned with filtered distilled water (0.45 μm Millipore membrane filter) prior to use.

2.3. Microplastic analysis

The surface seawater samples from the twelve marine stations were found to be rich in plankton (see Fig. 2). According to the study conducted by Quigg et al. (2013), plankton biomass and biodiversity in the NE coast of Qatar is higher than other parts of the Arabian Gulf. In the laboratory, samples were examined visually using a 3D stereomicroscope with an actual viewing magnification of up to 80 \times (Zeiss SteReo Lumar.V12 with NeoLumar S 0.8 \times FWD 80 mm). Any non-biological material resembling plastic debris was removed using metal forceps, then sorted and identified based on morphological characteristics (size, shape, color), as prescribed by Hidalgo-Ruz et al. (2012). Collected microplastics were transferred to a petri dish, then photographed prior to polymer identification.

Identification of polymer type was performed using a Fourier Transform Infrared (FTIR; Nicolet iS10) spectrometer equipped with Attenuated Total Reflectance (Smart iTR Diamond Crystal Plate). The instrument was operated in scanning transmittance mode at a mid-IR range of 650–4000 cm^{-1} . A background scan was performed to eliminate CO₂ interference (approx. 2300–2400 cm^{-1}). Twenty scans were performed per microparticle at medium resolution. The microplastic polymer spectra was verified using the OMNIC Spectra Library, and only polymers with a matching spectra >60% were accepted (Lusher et al., 2013; Avio et al., 2015).



Fig. 2. Example of the collected plankton-rich seawater samples (left-right: Stations D7, S5, D4).

Table 1
Number and polymer types of microplastics in seawater from the NE of Qatar's Exclusive Economic Zone (EEZ).

| Stations | Coordinates | Number of microplastics present | Volume of seawater (m ³) | Microplastic abundance (no./m ³) | Type of polymer |
|----------------|------------------------------|---------------------------------|--------------------------------------|--|--|
| D1 | N 25° 38.94' E 52° 12.87' | 1 | 4.96 | 0.20 | Polymethyl methacrylate |
| D2 | N 25° 37.61' E 52° 17.60' | 1 | 4.36 | 0.23 | Cellophane |
| D3 | N 25° 32.19' E 52° 15.84' | 3 | 2.65 | 1.13 | Polypropylene |
| D4 | N 25° 33.21' E 52° 08.92' | 3 | 2.67 | 1.12 | Polypropylene; poly(styrene:vinylidene chloride) |
| D5 | N 25° 34.80' E 52° 03.68' | 6 | 2.00 | 3.00 | Polymethyl methacrylate; polypropylene; low density polyethylene ; polystyrene; poly(ethylene:propylene:diene) |
| D6 | N 25° 24.34' E 52° 08.49' | 5 | 4.31 | 1.16 | Polyamide; cellophane; poly(styrene:vinylidene chloride); poly(ethylene:propylene:diene) |
| D7 | N 25° 34.96' E 52° 10.67' | 2 | 2.16 | 0.93 | Acrylonitrile butadiene styrene |
| S4 | N 25° 31.73' E 52° 13.18' | 1 | 2.26 | 0.44 | Cellophane |
| S5 | N 25° 35.51' E 52° 38.37' | – | 3.34 | 0.00 | – |
| Halul Island | N 25° 39.95' E 52° 26.07' | 7 | 3.48 | 2.01 | Polyethylene; polypropylene; low density polyethylene |
| Doha Channel | N 25° 17.81' E 51° 43.61' | – | 4.95 | 0.00 | – |
| Exit (Buoy #1) | N 25° 17.00' E 51° 42.76' | 1 | 4.85 | 0.21 | Cellophane |

3. Results and discussions

3.1. Results of method validation: sample digestion

Different digestion treatments were investigated and adapted from the studies of Cole et al. (2014) and Claessens et al. (2013). Cole et al. (2014) reported that the use of 1 M NaOH at room temperature proved 90% effective in digesting marine biota, and that by increasing the concentration and temperature to 10 M NaOH and 60 °C, digestion of plankton-rich samples was enhanced. Claessens et al. (2013) validated the use of 16 M HNO₃¹ at 100 °C for efficiently digesting bivalve mussel tissues for the extraction of microplastics.

As this study represented a primary investigation for Qatar, digestion treatments for extraction of microplastics for seawater samples were validated and optimized using 20 mL of 1 M NaOH, 10 M NaOH, and 16 M HNO₃. These solutions were spiked with known quantities of polymer pellets, as well as some commercially available plastic polymers (See Section 2.3). Extraction rates with solutions containing 10 M NaOH and 16 M HNO₃ resulted in structural damage and discoloration in some reference polymers. Polyvinyl alcohol (PVA) and polyvinyl chloride (PVC) were discolored after digesting in 10 M NaOH solution. Polyethylene terephthalate (PET) and polystyrene (PS) disintegrated in 16 M HNO₃, while PVC and nylon dissolved in 16 M HNO₃. The digestion method resulting in the highest particle recovery efficiency was 1 M NaOH (ANOVA, Tukey HSD post-hoc analysis $p < 0.05$), where recovery ranged from 98.52 ± 0.73% to 99.62 ± 0.27%. The least effective treatment was 16 M HNO₃ where recovery ranged from 37.72 ± 5.55% to 97.47 ± 0.71%, followed by 10 M NaOH ranging from 56.03 ± 2.26% to 98.96 ± 0.48%.

The three digestion treatments were also used to digest the plankton biomass. A concentrated solution of 10 M NaOH and 16 M HNO₃ in conjunction with high temperatures have been proven to mineralize biological materials i.e. plankton. In the study, both solutions proved to be 84.40 ± 0.84% and 94.23 ± 0.94% effective in digesting the plankton biomass respectively. The less concentrated 1 M NaOH solution was able to obtain 73.87 ± 2.73% digestion. Based on the optimization of the

microplastic extraction method for seawater, although 10 M NaOH and 16 M HNO₃ were effective at removing biological materials, these solutions also resulted in structural damage and dissolution of polymers with a low pH tolerance. All samples containing homogenized plankton biomass were then subjected to 1 M NaOH digestion treatment. After digestion, the samples were cooled to room temperature and vacuum filtered through 11 µm Whatman Filter paper (#1). Filtration using smaller pore size was not possible due to excessive clogging of the filter paper. The digested plankton biomass was then microscopically inspected for any presence of microplastic particles and analyzed for polymer type.

3.2. Microplastics in plankton-rich seawater samples

Seawater samples from ten out of the twelve marine monitoring stations had microplastics present (Table 1). In total 30 microplastic polymers have been identified. The microplastic concentration ranges from 0 to 3 particles m⁻³ (average concentration ~0.71 particle m⁻³). This incidence is considered low as compared to concentrations observed in the Northeast Pacific Ocean which has microplastic concentrations that range from 8 to 9200 particles m⁻³ (Desforges et al., 2014), and the Northeast Atlantic Ocean that has an average microplastic concentration of 2.46 ± 2.43 particles m⁻³ (Lusher et al., 2014). Reports of low microplastic concentration have been reported for the coastal waters of California, USA with average concentration ranges from 0.004 to 0.19 particles m⁻³ (Doyle et al., 2011); and in the Tamar Estuary, Southwest England, with an average concentration of 0.040 particles m⁻³ (Sadri and Thompson, 2014). Overall, the results from this study are comparable to those reported in the literature for coastal waters, and confirms the hypothesis that microplastics are ubiquitous in the marine environment of Qatar's EEZ.

The most numerous type of synthetic polymer identified in the study, from the ten stations where microplastics were present, was polypropylene (PP) which represented 9 out of the 30 microplastic particles collected. Polypropylene's resistance to high temperatures, wide-application and relatively low cost renders its prolific use in a wide range of consumer products (Lefteri, 2003), and is thus often the predominant polymer found in the environment. The chemical structure of polypropylene has distinct spectral bands for CH, CH₂ and CH₃ functional groups with peaks occurring at wave numbers 2848–

¹ L. Van Cauwenberghe, personal communication, February 11, 2016 - Concentration corrected from the previously published 22.5 M to 16 M HNO₃ (69% assay) by the corresponding author.

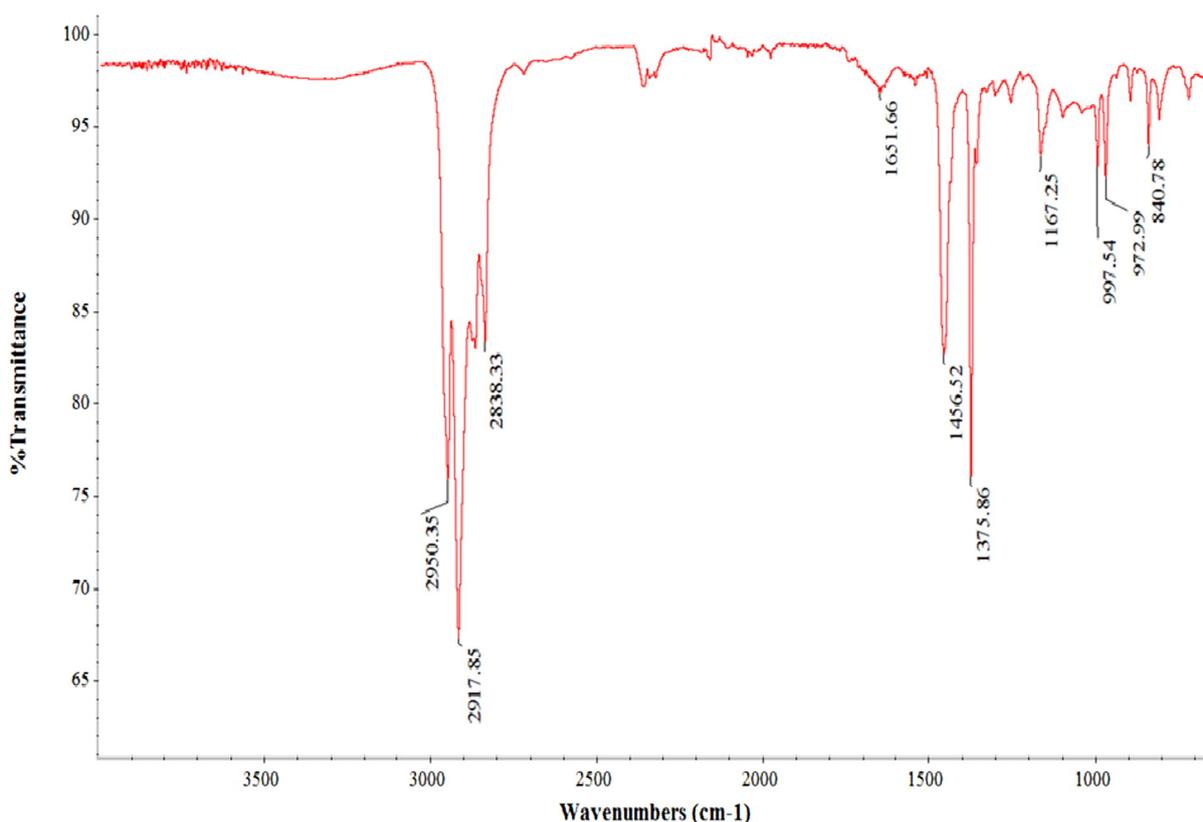


Fig. 3. FTIR spectra for a polypropylene microplastic particle collected from station D4.

2960 cm^{-1} , 1450–1470 cm^{-1} , 1375–1380 cm^{-1} respectively. As shown in Fig. 3, these peaks were evident in the spectra for the sample analyzed (Station D4). Moreover, the spectral bands observed at 840–1167 cm^{-1} were inherent for isotactic polypropylene bands. Generally, peaks between 700 and 770 cm^{-1} occur due to the rocking of the CH_2 groups which serve to distinguish between polypropylene and polyethylene. There is no distinct spectral band in this area for polypropylene, which confirms the identity of polypropylene polymer, as verified via the

OMNIC Spectra Library (Fig. 4). The weak spectral band around 1650–1700 cm^{-1} distinguishes the carbonyl group ($\text{C}=\text{O}$), which indicates oxidation of the polypropylene. Exposure to the marine environment and sunlight are major factors affecting degradation of plastic polymers (Barbeş et al., 2014). Qatar's arid, warm climate is likely to facilitate oxidation of primary plastic litter in its marine environment.

A number of other polymer types were isolated and identified from the seawater samples including: low density polyethylene (LDPE);

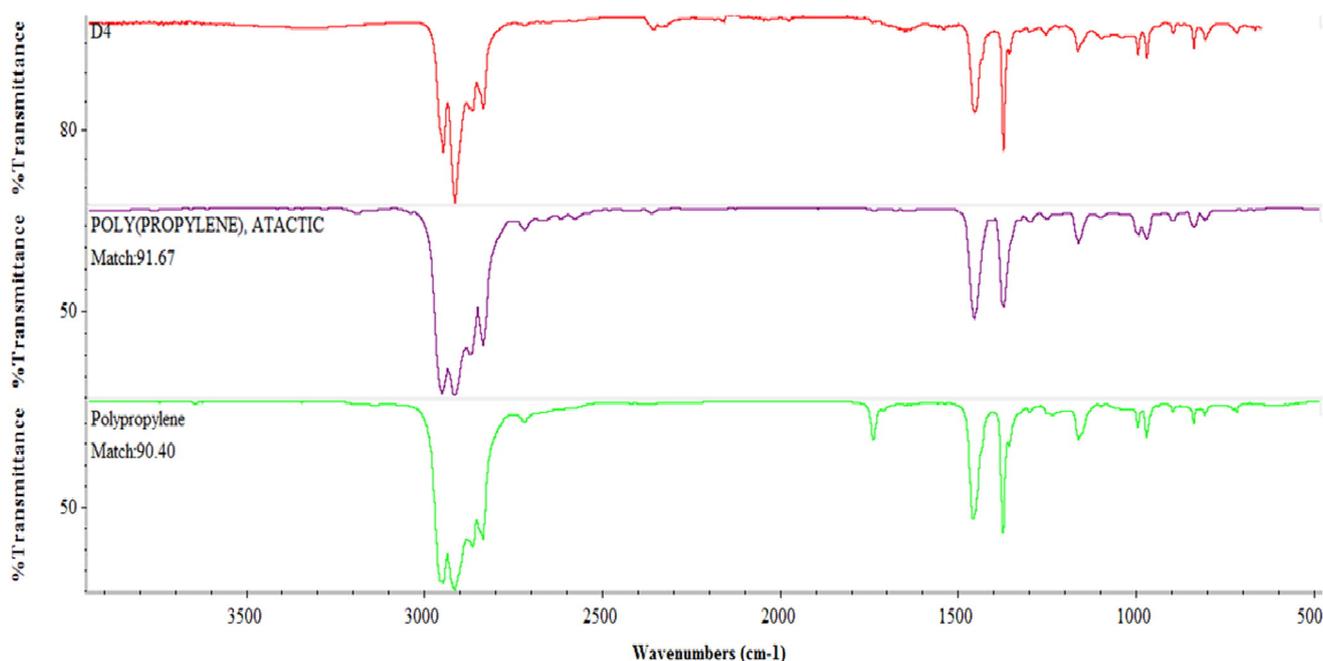


Fig. 4. FTIR spectra from the sample with the reference polypropylene spectra from Omnic Spectra library.

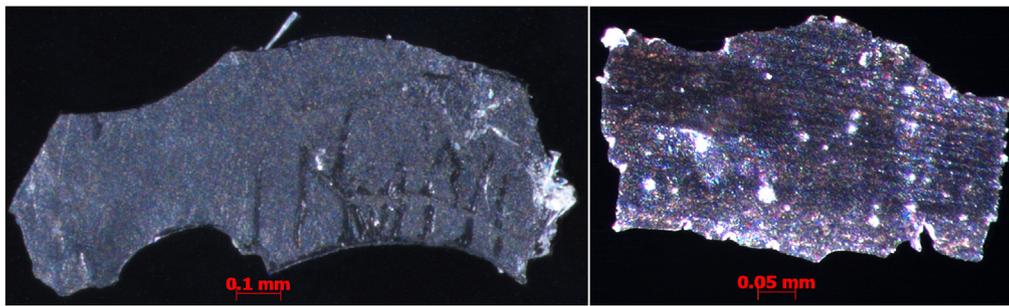


Fig. 5. Copolymer microplastic particles isolated from the marine waters of Qatar's EEZ. (left-right: poly(ethylene:propylene:diene) and poly(styrene:vinylidene chloride)).

polyethylene (PE); polystyrene (PS); polyamide (PA); acrylonitrile butadiene styrene (ABS); polymethyl methacrylate (PMMA); and cellophane microplastic particles. Copolymers and alkyd resin were also found in the collected samples, and can possibly be linked to fugitive losses from ship hulls, and ballast water tanks. Copolymers identified included poly(ethylene:propylene:diene) and poly(styrene:vinylidene chloride) which are known components of synthetic rubber (e.g. tires, conveying belts, electrical insulates) and lacquer coatings (e.g. interior

coatings for ship tanks, fuel storage tanks and coating for steel pipes) (Riyaz and Desai, 2013; Chen et al., 2015; Othmer, 1997; see Fig. 5).

The majority of the microplastic polymers identified in the seawater samples from the Qatar EEZ were either granular or fibrous in shape. Granular shaped microplastics ranged from 125 μm to 1.82 mm in size, and fibrous microplastics from 150 μm to 15.98 mm. The colors of the particles varied widely, but blue and opaque white were the most common (Fig. 6; Supplementary Tables 1 and 2). The greatest

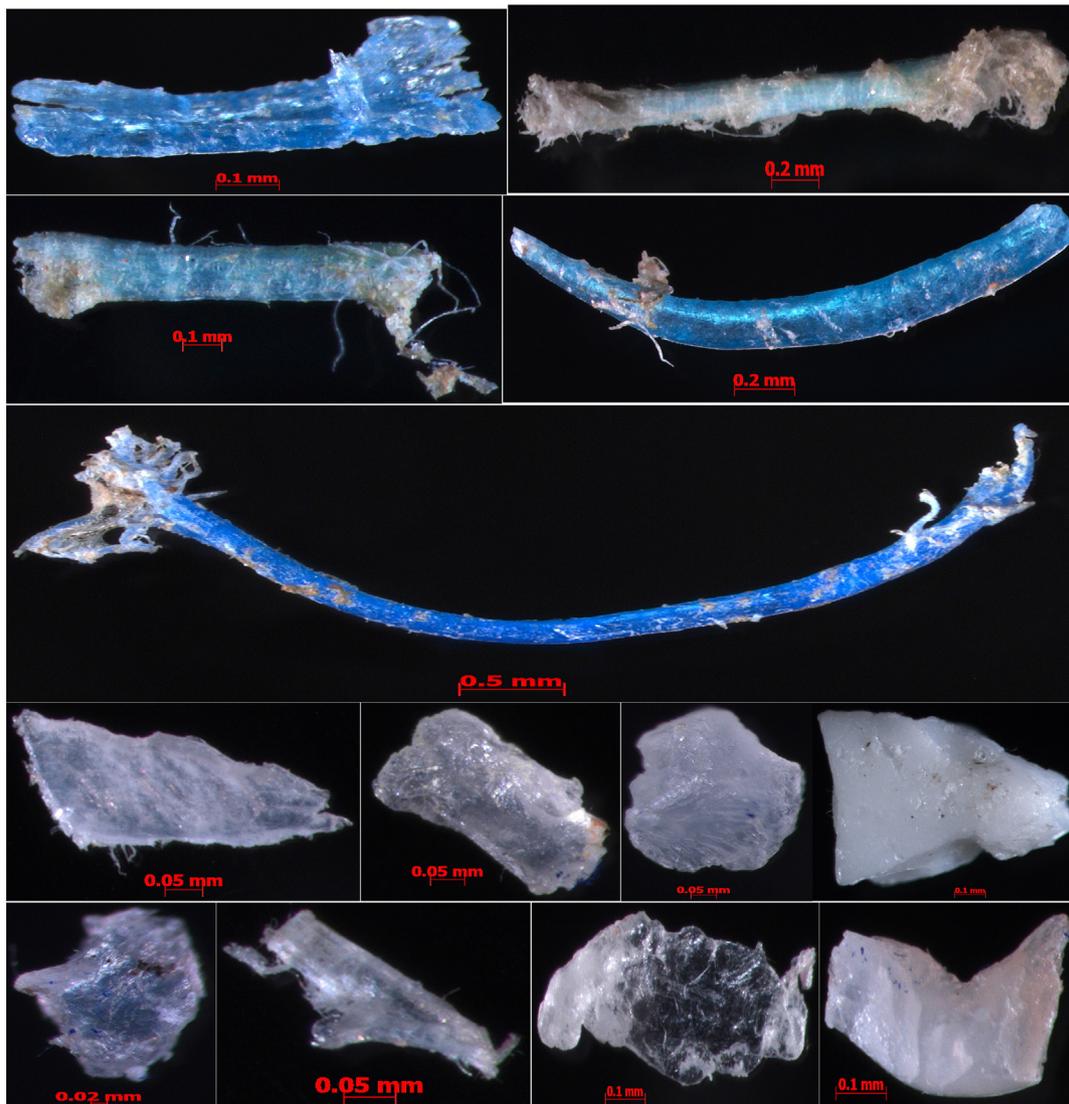


Fig. 6. Microplastic particles isolated from the seawater samples in the NE marine waters of the Qatar's EEZ.

concentration of microplastics was found to adjacent Halul Island in the northeast of Qatar's EEZ (see Fig. 1), where nearby anthropogenic activities include oil-rig installations and shipping operations.

4. Conclusions

Spatial variation of microplastic concentration in the northeastern marine waters of the Qatar EEZ was apparent, and was influenced by proximity of nearby sources of anthropogenic activities. The concentration of microplastics occurring in surface seawater ranges from 0 to 3 particles m^{-3} with an average concentration of 0.71 particle m^{-3} . Several polymer types were isolated and identified, including copolymers. Copolymers and alkyd resins found were possibly linked to fugitive losses from ship hulls and ballast water tanks. The microplastic concentration observed in this study is considered low in the context of the reported microplastic concentrations in seawater from the studies of Desforges et al. (2014) and Lusher et al. (2014), but comparable to the results observed in the studies of Doyle et al. (2011) and Sadri and Thompson (2014). Overall, the results from this study confirms the hypothesis that microplastics are ubiquitous in the marine waters of Qatar.

Extraction of microplastics in the plankton-rich waters of Qatar proved challenging. However, an optimized digestion procedure using a 1 M NaOH alkaline solution was developed for the efficient extraction of microplastics from the seawater samples. Microplastics extracted were in the class of lower density plastics e.g. PP, PE, LDPE, PA, ABS. Microplastics found in Qatar's EEZ showed evidence of oxidation, as based on the presence of carbonyl groups in the FTIR analysis. The presence and intensity of the spectral band specific for carbonyl groups is an indicator of plastic degradation, where exposure to the high salinity of Qatar's marine environment, as well as elevated levels of ultraviolet radiation, create an aggressive environment for the degradation of plastic debris into microplastics.

This investigation on the prevalence of microplastics in Qatar's marine EEZ will be extended to include marine sediments and biota in future studies, so as to facilitate our understanding of source identification of microplastics, and to identify the potential for microplastic-associated pollutant transfer in Qatar's marine food chain.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.marpolbul.2016.06.108>.

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