Environ Geochem Health (2021) 43:3733–3748
https://doi.org/10.1007/s10653-021-00872-8

ORIGINAL PAPER

Occurrence, abundance, and distribution of microplastics pollution: an evidence in surface tropical water of Klang River estuary, Malaysia

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Received: 6 August 2020 / Accepted: 27 February 2021 / Published online: 12 March 2021

Abstract Microplastics have been considered as contaminants of emerging concern due to ubiquity in the environment; however, the occurrence of microplastics in river estuaries is scarcely investigated. The Klang River estuary is an important ecosystem that receives various contaminants from urbanised, highly populated areas and the busiest maritime centre in Selangor, Malaysia. This study investigates the abundance and characteristics of microplastics in surface water of the Klang River estuary. The abundance of microplastics ranged from 0.5 to 4.5 particles L^{-1} with a mean abundance of 2.47 particles L^{-1}. There is no correlation between the abundance of microplastics and physicochemical properties, while there is a strong correlation between salinity and conductivity. The microplastics were characterised with a stereomicroscope and attenuated total reflection–Fourier transform infrared spectroscopy to analyse size, shape, colour, and polymer composition. The microplastics in the surface water were predominantly in the 300–1000 μm size class, followed by > 1000 μm and < 300 μm, and were mostly transparent fibres, fragments, and pellets. Polyamide and polyethylene were the main polymer types in the composition of the microplastics, suggesting that the microplastics originated from heavily urbanised and industrial locations such as the port, jetty, and residential areas. The widespread occurrence of microplastics in the environment and subsequent penetration of aquatic food webs may pose a serious threat to organisms. This study provides baseline data and a framework for further investigation of microplastic contamination in estuaries.

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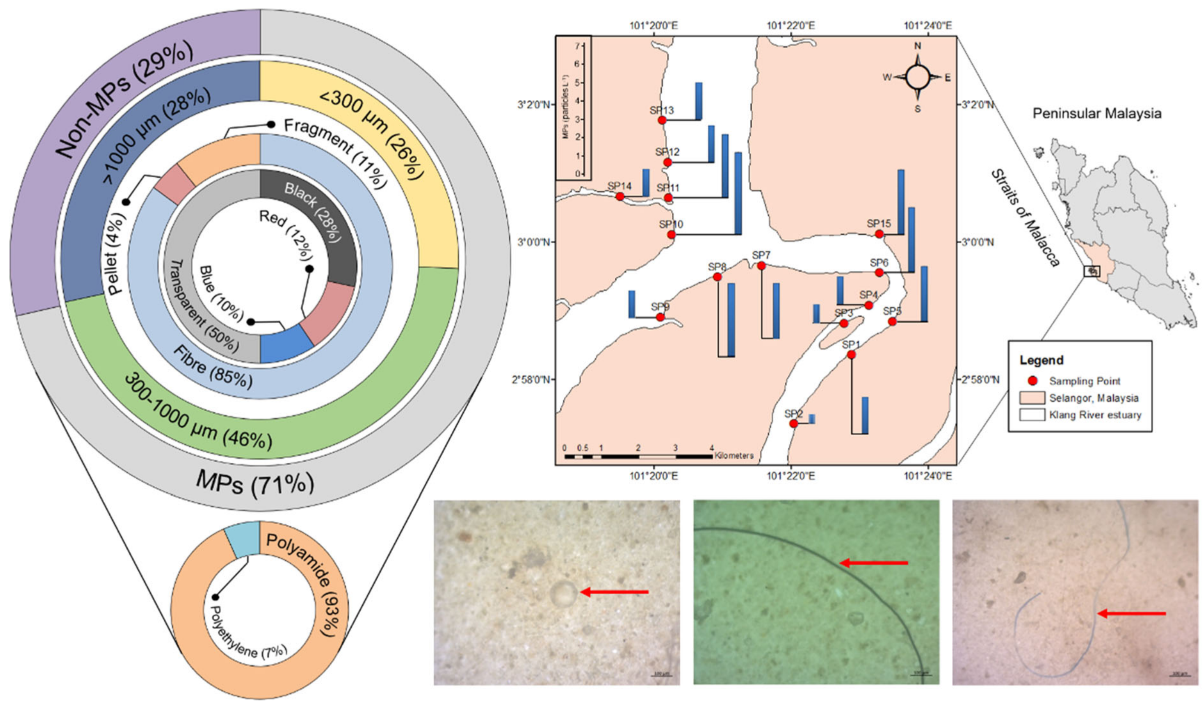
Graphical abstract



Keywords Microplastics · Klang River estuary · Surface water · Microfibre · Anthropogenic activities · Baseline study

Introduction

Plastics have become an indispensable part of life due to the unique properties such as lightweight, durability, and convenience. Extensive mass production of plastics has led to an estimated 4.8–12.7 million tonnes of plastic waste being released into the ocean (Jambeck et al., 2015). Microplastics (MPs) are defined as particles less than 5000 µm (5 mm) in size (Thompson et al., 2009) and are classified into primary and secondary MPs (Cheung and Fok, 2016). Primary MPs are microsized plastics manufactured by industry, while secondary MPs are smaller particles formed by fragmentation or degradation of plastic through physical (e.g. wind, wave and tidal abrasion), chemical (e.g. adsorption of an organic pollutant or heavy metal), and biological (e.g. biofilm formation) processes in the environment (Cole et al., 2011).

The physical characteristics, chemical composition, surface area, and hydrophobic characteristics of the particles, as well as the pH and salinity of the environment, are strongly associated with the abundance of MPs in the environment (Liu et al., 2019). Owing to small size, abundance, and ability to float in aquatic ecosystems, MPs are mistaken for food particles and ingested by aquatic organisms. In addition, nanoplastics, with particle size (diameter) ranging from 1000 to 1 µm, may enter the digestive tract passively with water (Chae and An, 2017; Jiang et al., 2020). Ingestion of and entanglement in MPs are widely reported, causing an ecological and human health implications (Abidli et al., 2019; Nelms et al., 2019). The adsorption of organic pollutants from the environment is attributed to the large surface area of MPs (Cole et al., 2011). In addition, previous studies have shown that nanoplastics have a higher tendency to carry organic pollutants to the environment than MPs due to the large surface area of the particles (Velzeboer et al., 2014; Brandts et al., 2018). This contributes to the bioavailability of plastic particles and organic pollutants simultaneously in the environment. Water quality parameters, such as pH, temperature, salinity, dissolved oxygen, biochemical oxygen demand (BOD) and turbidity, have been shown to influence the occurrence of MPs and organic pollutants in the environment. A previous study of a river basin in Japan demonstrated a significant relationship between MP concentration and BOD, suggesting that the lack of a sewerage system contributed to poor water quality (Kataoka et al., 2019). The concentration of MPs tends to increase with turbidity level, which may be related to the impacts of estuary dynamics, urbanisation, and poor wastewater treatment (Pazos et al., 2018). On the other hand, other parameters including salinity, temperature, and pH are mostly related to adsorption, desorption, and leaching of persistent organic pollutants (POPs) from MPs (Bakir et al., 2014; Liu et al., 2019). Exposure to POPs is reported to impair the reproductive system and sexual function and is linked to DNA damage, endometriosis, polycystic ovary syndrome, and breast cancer in humans (Tarantino et al., 2013). Plastics have numerous chemical additives, such as plasticisers, antioxidants, flame retardants, and dyes, to enhance durability. These chemicals are eventually leached out and pollute aquatic ecosystems (Jiang et al., 2019).

MPs are perceived to be an alarming issue and global pollution due to the ubiquity in the environment and possess a detrimental impact on ecosystems. High economic growth, industrialisation and urbanisation, as well as improper waste management, lead to MP contamination of aquatic environments (Pazos et al., 2018; Piehl et al., 2019). Estuary and coastal environments have specifically been indicated as hot spots of MP pollution. These areas are heavily populated and affected by various anthropogenic activities such as industrialisation and urbanisation (Yonkos et al., 2014; Silva-Cavalcanti et al., 2017). Moreover, estuaries receive an abundance of marine and terrestrial litter due to the strategic location linking land and sea (Yan et al., 2019; Zhang et al., 2019). Estuaries consist of various habitats including mangrove and are important for reducing coastal erosion by slowing down water flows and encouraging sediment deposition. Mangroves act as nursery grounds and provide habitats for marine birds, mammals, fishes, and crustaceans. Numerous studies have reported MP contamination in estuarine organisms such as fish (Pegado et al., 2018), crab (Waite et al., 2018), and oysters (Li et al., 2018; Waite et al., 2018).

The occurrence and distribution of MPs in riverine and marine ecosystems have gained worldwide attention. However, few studies have addressed the occurrence and potential sources of MPs in the surface water of the estuarine ecosystems of Malaysia, particularly in the tropical region. Khalik et al. (2018) addressed the MP contamination in a coastal area (Kuala Nerus) and a port area (Kuantan Port), both of which are in the east coast of Malaysia, facing the South China Sea. The study indicates various common types of MPs, including polystyrene (PS), polyethylene (PE), polyamide (PA), and polyvinyl chloride (PVC), in selected locations representing the presence of anthropogenic activities such as fishing and shipping. This study addresses the occurrence of MPs in the west coast of peninsular Malaysia with heavy marine traffic at Port Klang, maritime activity, and rapid industrialisation (Haris and Aris, 2013; Omar et al., 2019). Compared to the port area (Kuantan Port) studied by Khalik et al. (2018), Port Klang is the second busiest container port in Southeast Asia after the Port of Singapore and ranked the 12th busiest globally (Nguyen et al., 2020; World Shipping Council, 2021). This study may also reveal the occurrence of MPs in estuarine tropical waters, which have been recognised as a hot spot and sink for various pollutants. This study aims (i) to analyse the abundance and distribution of MPs in surface water from Klang River estuary, and (ii) to elucidate the physical characteristics of MPs present in surface water. The findings from this study will provide explanatory data for MPs in surface water and specifically address the occurrence and physical characteristics of MPs in urbanised estuary rivers.

Materials and methods

Study area

The Klang River and Langat River are important sources of freshwater for the residents of Selangor. The river runoff from distinct cities in Malaysia such as Kuala Lumpur, Shah Alam, and Petaling Jaya is discharged into the Klang River estuary. The Klang River basin is approximately 120 km long and is the main source of freshwater for more than four million people. The Langat River flows through urbanised and populated towns including Cheras, Bandar Baru Bangi, Kajang, and Putrajaya. The combined river runoff from the Klang and Langat rivers potentially influences the contamination of the water in the Klang River estuary. Along the rivers and the estuary region of Klang, multiple types of land use such as industrial, commercial, residential, and agricultural which contribute to the pollution of the Klang River estuary (Haris and Aris, 2013; Omar et al., 2018; Fang et al., 2019). Klang River estuary receives large quantities of anthropogenic waste from sewage treatment, marine traffic, and industrial, agricultural, and tourism activities (Mokhtar et al., 2015; Haris et al., 2017). In addition, Port Klang is the busiest and largest port in Malaysia and is a potential source of various pollutants in the estuary. Assessment of contaminants in the Klang River estuary has mostly focused on organic (Omar et al., 2018, 2019) and heavy metal contamination (Sany et al., 2013; Haris and Aris, 2013, 2015; Haris et al., 2017). There are few studies on the abundance and distribution of MPs in the surface water of estuarine rivers of Malaysia.

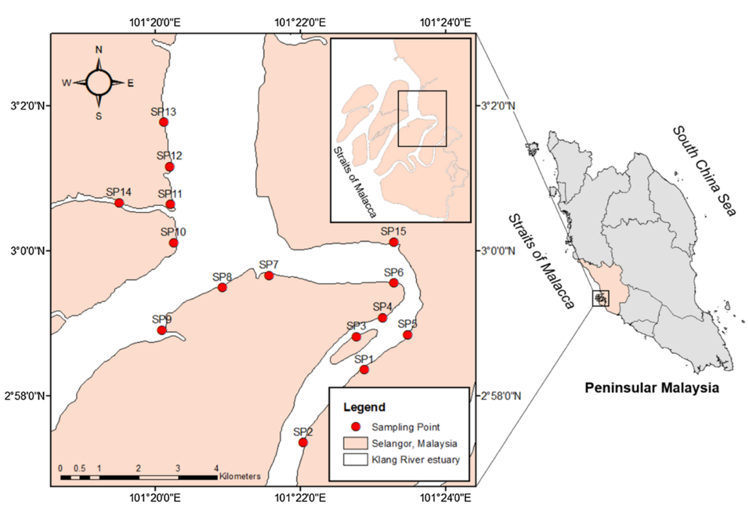
Sampling

Surface water samples were collected from 15 sampling points in various land use areas of the Klang River estuary in Selangor in September 2019 (Fig. 1a, b). The samples were collected by fully submerging 1-L glass bottles about 45 cm from the surface. Bubbles or air spaces were avoided to prevent cross-contamination. Samples were stored at 4 °C prior to experimental analysis. In situ parameters, such as temperature, pH, conductivity, salinity, turbidity and dissolved oxygen, were obtained using a calibrated portable multiprobe meter (YSI Pro Plus, YSI Inc., Yellow Springs, Ohio, USA) and a portable turbidimeter (Hach 2100P, HACH Lange GmbH, Dusseldorf, Germany).

Preparation and extraction of microplastic

MPs were prepared and extracted following the method described in Masura et al. (2015) with a slight modification. In brief, each water sample was passed through a 5.6-mm stainless steel sieve in the laboratory. The large particle residues were discarded prior to experimental analysis. The water sample was immediately treated with 20 mL of a mixture of 0.05 M iron (II) sulphate ($FeSO_4$; Fisher Scientific, UK) and 30% hydrogen peroxide (H_2O_2; Fisher Scientific, UK) solution (1:2, v/v) under a fume hood to reduce any organic material and airborne contamination. Prior to extraction, the mixture was heated to 75 °C on a hot plate to enhance rapid digestion of organic material. Once gas bubbles were observed, the beaker was immediately removed from the hot plate to allow the boiling to subside. Formation of bubbles in the reaction indicated physical changes in MPs. The mixture was heated for an additional 30 min until no natural organic material was observed. Prior to density

(a)



(b)

Sampling point	Coordinate	Land use area
SP1	N 2°58'21.6" E 101°22'52.4"	Jetty, mangrove and residential
SP2	N 2°57'21.6" E 101°22'01.8"	Mangrove and residential
SP3	N 2°58'49.0" E 101°22'45.7"	Mangrove
SP4	N 2°59'04.9" E 101°23'07.5"	Near mangrove area
SP5	N 2°58'50.8" E 101°23'28.0"	Jetty and industrial
SP6	N 2°59'33.3" E 101°23'16.6"	Mangrove and shipping lane
SP7	N 2°59'39.5" E 101°21'34.0"	Industrial and mangrove
SP8	N 2°59'29.3" E 101°20'55.1"	Recreational and residential
SP9	N 2°58'54.5" E 101°20'05.3"	Westport terminal and opposite to mangrove
SP10	N 3°00'06.6" E 101°20'15.3"	Mangrove and shipping lane
SP11	N 3°00'38.3" E 101°20'12.5"	Mangrove and opposite to Northport terminal
SP12	N 3°01'09.5" E 101°20'11.5"	Mangrove and Northport terminal
SP13	N 3°01'46.3" E 101°20'06.9"	Mangrove and Northport terminal
SP14	N 3°00'49.5" E 101°19'35.6"	Mangrove
SP15	N 3°00'19.9" E 101°23'17.6"	Industrial, jetty and residential

Fig. 1 Map with the **a** location of sampling points and **b** land use area present in Klang River estuary

separation, the mixture was added to a 5 M saline solution (NaCl; Merck, Germany) and transferred to a separating funnel following the method described by Masura et al. (2015). The supernatant was collected and filtered using 0.45-µm filter paper (Whatman sterile membrane filters, cellulose nitrate, 47 mm diameter, USA) under a vacuum system. Prior to observation of MPs, the filter paper was covered with aluminium foil and stored in a glass petri dish to minimise airborne contamination.

Visual identification of microplastic

MPs were observed using a stereomicroscope (ZEISS Axioskop 2, Germany), and images were captured with an AxioCam ERc 5 s digital camera (Zeiss, Germany). All physical characteristics, such as size, shape and colour, were observed through the microscope and analysed with ZEN 2.5 blue edition software (Zeiss). Furthermore, the polymer composition of MPs was identified using a Thermo Fisher Scientific microscope Nicolet 6700 Fourier transform infrared spectroscopy (FTIR) spectrometer attached with attenuated total reflection (ATR). Approximately 42 samples were randomly selected to analyse polymer composition. ATR-FTIR was operated on single reflection mode in the spectrum range of 4000–800 cm^{-1} with 4 cm^{-1} resolution and 32 scans throughout the analysis. Each sample spectrum was compared with reference spectra provided by the HR Nicolet Sampler Library (Thermo Fisher Scientific).

Statistical analysis

Abundance of MPs reported as particles L^{-1} and its mean and standard deviation (SD) were obtained. The relationships between MPs polymer composition and physicochemical properties were assessed using bivariate analysis. The differences were considered significant at $p < 0.05$. The statistical analyses were performed using IBM SPSS version 25 software (IBM, USA).

Quality assurance and quality control (QAQC)

Since there are no standardised techniques provide optimum efficiency while being cost-effective and less time-consuming, the surface water samples collected in this study were of small volume for reliability. Many studies have used water samples of large volume based on bulk sampling (e.g. Zhao et al., 2014; Wang et al., 2018; Yan et al., 2019) or plankton tows (Yonkos et al., 2014; Di and Wang, 2018). However, a large volume is not enough to guarantee samples representative of MPs occurrence and abundance in the environment. In addition, samples of large volume are costly and time-consuming to collect and have a high potential for loss of smaller MPs (Pico et al., 2019). McEachern et al. (2019) compared two different sampling techniques, discrete samples (1 L) and plankton tow samples (roughly 10,000 L), and showed that the discrete samples with 1 L of water sample acquired a better concentration of MPs compared to plankton tows samples. It was crucial to consider cost and time efficiency in this study while ascertaining the occurrence and abundance of MPs in estuarine water; therefore, the volume of both control and environmental samples was limited to 1 L of surface water with replicates, following the protocol of McEachern et al. (2019).

Recovery analysis was performed to evaluate the efficiency of the extraction method by using standard reference material as the internal standard. Optimum percentage recoveries (95–105%) were acquired for optimisation of MP extraction and analytical method based on Masura et al. (2015). In brief, a mixture of 0.05 g of granular homopolymer polypropylene (PP; US Pharmacopeia, CAS number 9003-07-0) and low-density polyethylene (LDPE; European Reference Materials, CAS number 9002-88-4) was spiked to synthetic estuarine water. Synthetic estuarine water was prepared by following method described in Omar et al. (2019). The extraction recovery of the internal standard was evaluated using the synthetic estuarine water to simulate actual estuarine water conditions. As prior quality control, the procedural blank sample ($n = 3$) was included in the extraction method for both the control and environmental sample. Samples at each sampling point were collected in replicates and glassware was precleaned using Decon 90. Consequently, deionised water was used to rinse the glassware and oven-dried for 24 h at 65 °C. In order to mitigate and minimise cross-contamination of the samples, solutions and solvents, such as deionised water, MeOH, $FeSO_4$, and H_2O_2, were prefiltered through a 0.45-µm filter during sample preparation. This precautionary step is crucial as particles coexisting in solutions or solvents may be mistaken for MPs from the samples.

Results and discussion

Abundance and distribution of microplastic

The abundance and distribution of MPs were evaluated for 15 sampling points in the Klang River estuary, which covered various urban areas and ecosystem types, e.g. port, residential, industrial, forest, and mangrove areas. MPs were present in surface water samples at all sampling points in the Klang River estuary. In total, 74 particles were observed with an average abundance of 2.47 ± 1.19 particles L^{-1} (Fig. 2). MPs varied across the sampling points (SP), with SP10 showing the highest abundance (4.50 ± 2.12 particles L^{-1}), while SP2 had the lowest abundance (0.50 ± 0.71 particles L^{-1}). The high abundance of MPs in this study may be attributed to the disposal of domestic waste, and anthropogenic activities may affect the abundance and occurrence of MPs. Both SP10 and SP8 (4.00 ± 1.41 particles L^{-1}) were located near the port and areas of anthropogenic activity, which suggests that the occurrence of MPs is predominantly influenced by the activities of the port and industrial and residential areas and fishing (Fig. 1b). Sampling points with low MP abundance, such as SP2 (0.50 ± 0.71 particles L^{-1}) and SP3 (1.00 ± 1.41 particles L^{-1}), were in proximity to the mouth of the Langat River and the mangrove area and were far from anthropogenic activities. In SP4 (1.50 ± 0.71 particles L^{-1}), SP9 (1.50 ± 0.71 particles L^{-1}) and SP14 (1.50 ± 2.12 particles L^{-1}), MPs were less abundant than in SP2 and SP3 due to the proximity of the sampling points to the mangrove area. This indicates that the low MP abundance is attributable to the smaller population in this area (i.e. forested or mangrove area) which received relatively low amounts of pollutants (Yonkos et al., 2014).

Despite the low MP abundance in the upper estuary (at SP2), the increased MP abundance at SP1 and SP5 appears to be related to the presence of the jetty, homes, and industries. In fact, MP occurrence has been attributed to the presence of jetties and fishery activities in the Jinhae Bay, south-eastern Korea (Song et al., 2015), Guanabara Bay, Brazil (Castro et al., 2016), and the coastline of Singapore (Curren and Leong, 2019). In addition, SP1–SP5 (except SP2) were located within the area where freshwater from the Langat River and seawater from the Straits of Malacca are mixed. Haris and Aris (2015) showed that the increased metal concentration in the area of seawater–saltwater mixing is due to the sedimentation occurring upstream of the Langat River, which thus influences the variation in pollutant concentration. Similarly, the variation in MPs in SP1–SP5 in this study shows the differential impact of sedimentation, riverine influences, and water mixing on the abundance and distribution of MPs (Frère et al., 2017; Dris et al., 2018; Song et al., 2018). SP2 was located closer

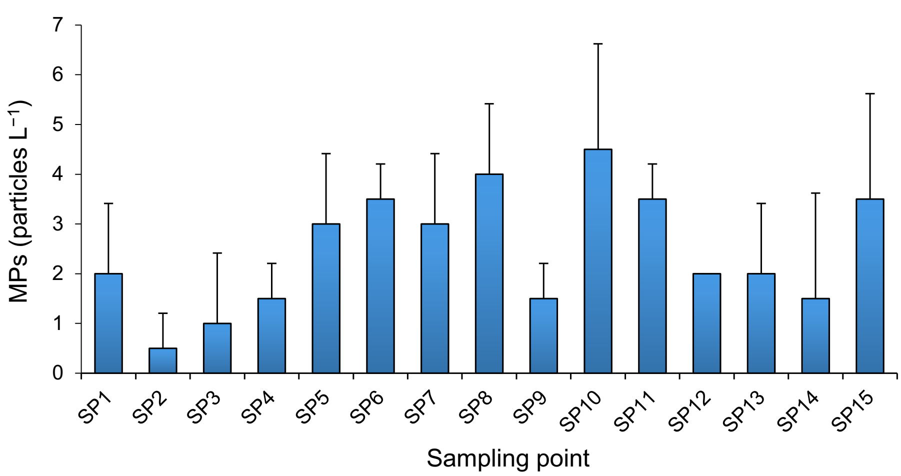


Fig. 2 Mean abundance of MPs in surface water across 15 sampling points of Klang River estuary. Error bars indicate ± standard deviation from experimental replicate

to freshwater, without seawater intrusion and anthropogenic influences, which suggests that it should have the lowest deposition of pollutants from both the Klang River estuary and the Langat River. However, MP abundance at SP2 was high due to the discharge of water from the Langat River which flows through cities, including Kajang, Bandar Baru Bangi, Putrajaya, and Cheras. However, a better understanding of transportation of MPs from upstream to downstream is needed with consideration of factors such as the duration of particle transport, water velocities, and distance of river.

The high occurrence of MPs indicates direct sources from various commercial activities and the main route for boats and vessels (Omar et al., 2018). Shipping and fishing activities contribute to the occurrence of MPs in nonurbanised and less densely populated areas (Lusher et al., 2015). Intense anthropogenic and land use activities contributed to the MP pollution of Hong Lake, China (Wang et al., 2018), and Patapsco River, USA (Yonkos et al., 2014). Since there are no standardised techniques for MP sampling, surface water samples were collected in different ways in previous studies. Therefore, this study was compared to previous studies that collected surface water samples using similar methods (Table 1). The abundance of MPs in the Klang River estuary is higher than that in Tampa Bay (0.94 ± 0.52 particles L^{-1}) (McEachern et al., 2019) and lower than that in Ciwalengke River, Majalaya district (5.85 ± 3.28 particles L^{-1}) (Alam et al., 2019). However, the MP abundance of small estuaries in Shanghai was higher (27.84 particles L^{-1}) than the abundances in this study due to the dynamics of small-scale rivers with high water velocity (Zhang et al., 2019). In addition, compared to an open-water estuary like Tampa Bay, the Klang River estuary is semi-closed with various anthropogenic wastes being introduced directly. In contrast, the Ciwalengke River surface water samples were collected in informal settlements, industrial and densely populated areas that contributed to the higher abundance of MPs. Furthermore, the abundance of MPs in the Pearl River Estuary (Yan et al., 2019), Yangtze Estuary (Zhao et al., 2014) and Minjiang (Zhao et al., 2015) was higher compared to the abundances in this study. With highly populated areas and various anthropogenic activities, the Pearl River located nearby to Guangzhou, one of the megacities of China, produced almost 10 million tons of plastics in 2016 (Yan et al., 2019).

Moreover, environmental conditions, such as pH, salinity and temperature, and physical abrasion due to tides, waves and currents contribute to the variations in the distribution of MPs in surface water (Zhang et al., 2019). In this study, salinity was significantly correlated with conductivity ($r = 0.920$, $p < 0.01$), while there were no correlations between physicochemical properties and MP abundance (Table 2). The pH of the surface water of the Klang River estuary ranged from 7.05 to 7.72. This is due to the waste generated by different land use activities, such as transportation, industry and fishing, in the Klang River estuary and residential areas along the Langat River (Haris et al., 2017). The highest temperature was found at SP7 (24.92 °C), which was located closer to the main commercial shipping route, port, and industrial area (Omar et al., 2018). Salinity and conductivity of the surface water of the Klang River estuary were in the range of 9.37–34.45 ppt and 16,092–52,328 µS cm^{-1}, respectively. In general, salinity increases with conductivity due to the presence of mineral salts in sediment which acts as a conductor of electricity (Shafie et al., 2013; Haris et al., 2017). The low salinity of surface water in SP2 and SP15 shows that the sampling points located in the upper estuary were closer to the freshwater environment. However, the abundance of MPs between SP2 and SP15 gave a contradictory result as SP15 tended to be exposed to and persistently received urban discharge (e.g. domestic and industrial effluents) from the Klang and Langat Rivers, thus causing it to have higher MPs abundance. Sampling stations located towards the lower estuary area tend to experience seawater exposure or intrusion, consequently resulting in greater salinity and higher pH (Lim et al., 2012; Cheung et al., 2018). This can occur in the mixing area or high-water-velocity areas (Haris and Aris, 2013), especially at SP15 and SP6, with saltwater intrusion and urban runoff from the Klang River. Therefore, the complex circulation pattern and the dynamics of coastal processes influence the input and distribution of MPs in the Klang River estuary. Vertical mixing, water flow, and deposition of sediment are greatly contributing to the transport and distribution of MPs in water bodies (Zhao et al., 2015; Nizzetto et al., 2016).

Furthermore, coastal areas act as sinks for environmental and emerging pollutants including MPs (Nel

Table 1 Comparison of MPs abundance (mean and range) with dominated MPs characteristics observed in estuarine water of Langat River estuary to other locations

Location	MPs abundance		Dominant MPs characteristic (%)				Reference
	Mean	Range	Size (µm)	Shape	Colour	Polymer composition	
Pearl River Estuary, China	8902 particles m^{-3}	7850– 0,950 particles m^{-3}	500 – 2000 (n/s)	Granule (48%) Film (43%)	n/s	n/s	Yan et al. (2019)
Yangtze Estuary, China	4137.3 particles m^{-3}	500– 0,200 particles m^{-3}	500 – 1000 (67%)	Fibre (79.1%)	Transparent (58.9%)	–	Zhao et al. (2014)
Minjiang, China	1245.8 particles m^{-3}	100– 100 particles m^{-3}	500 – 5000 (> 90%)	Fibre (n/s) Granule (n/s)	Black (n/s) Transparent (n/s)	PP (51.2%)	Zhao et al. (2015)
Jiaojiang, China	955.6 particles m^{-3}						
Oujiang, China	680.0 particles m^{-3}						
Shanghai, China	27.84 particles L^{-1}	13.53– 4.93 particles L^{-1}	≤ 2000 (99.5%)	Granule (38.04%) Fragment (35.57%) Film (22.52%)	Black (56.46%)	PE (50%) PP (37.5%)	Zhang et al. (2019)
Tampa Bay, US	0.94 particles L^{-1}	0.25– 7.0 particles L^{-1}	–	Fibre (76%)	–	–	McEachern et al. (2019)
	4.5 particles m^{-3}	1.2– 18.1 particles m^{-3}	–	Fibre (88%)	–	–	
Klang River estuary, Malaysia	2.47 particles L^{-1}	0.5 – 4.5 particles L^{-1}	300 – 1000 (46%)	Fibre (85%)	Transparent (50%)	Polyamide (67%)	This study

PP, Polypropylene; PE, Polyethylene; n/s, not stated

Table 2 Correlation coefficient between polymer composition of MPs and physicochemical properties in surface water of Klang River estuary

	PA	PE	pH	Temp	Sal	Cond	DO	Turb
PA	1.000							
PE	− 0.262	1.000						
pH	0.262	0.050	1.000					
Temp	0.144	0.341	0.767**	1.000				
Sal	0.377	0.038	0.462	0.226	1.000			
Cond	0.434	− 0.079	0.549*	0.247	0.920**	1.000		
DO	0.295	− 0.264	− 0.152	− 0.568*	0.460	0.483	1.000	
Turb	− 0.020	0.001	− 0.092	0.261	− 0.559*	− 0.461	− 0.614*	1.000

PA, Polyamide; PE, Polyethylene; Temp, Temperature; Sal, Salinity; Cond, Conductivity; DO, Dissolved oxygen; Turb, Turbidity

*Correlation is significant at the 0.05 level (2-tailed)

**Correlation is significant at the 0.01 level (2-tailed)

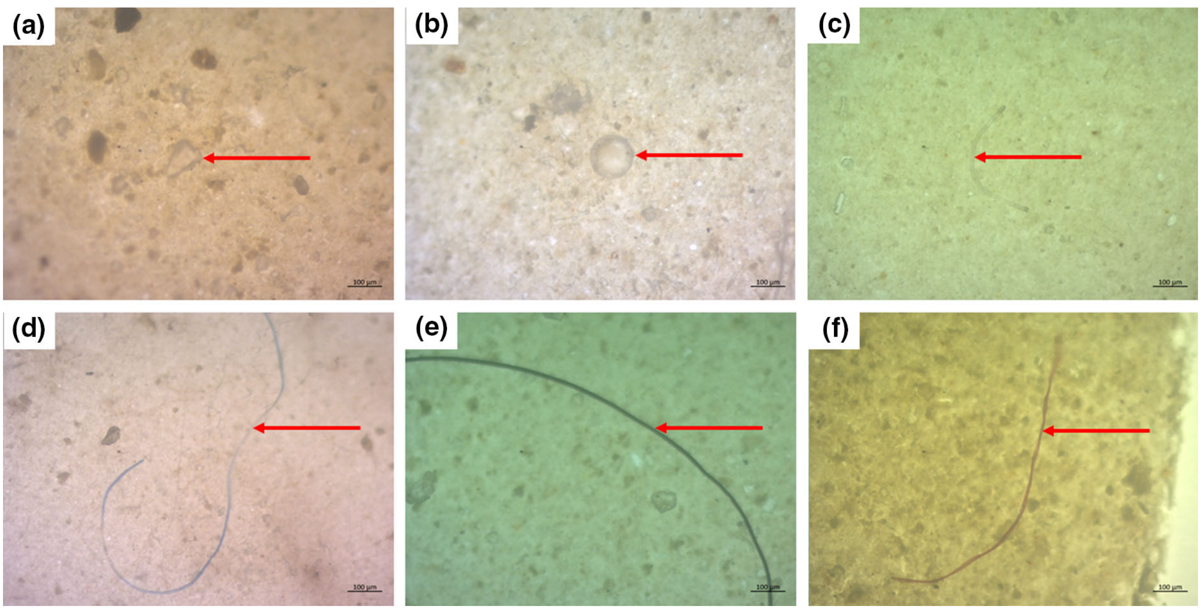


Fig. 3 Microscopic images of suspected MPs from surface water, consist of **a** transparent fragments, **b** transparent pellets, **c** transparent fibre, **d** blue fibre, **e** black fibre, and **f** red fibre

et al., 2018; Liu et al., 2019). This is because coasts and estuaries receive different types of waste from both maritime and landward activities. Zhao et al. (2015) showed that MPs are introduced into estuaries and coastal areas by river runoff and originate from landward activities. Thus, MPs continue to drift out to the open sea depending on the dynamics of coastal processes (Nizzetto et al., 2016). Nonetheless, wastes from populated and heavily urbanised areas contribute to the ubiquity of MPs and other organic pollutants in rivers, estuaries, coastal areas, and the open sea (Yonkos et al., 2014).

Microplastic characteristics

MPs were observed and images obtained using a stereomicroscope at 10 × magnification and attached camera, respectively (Fig. 3). A total of 74 identified

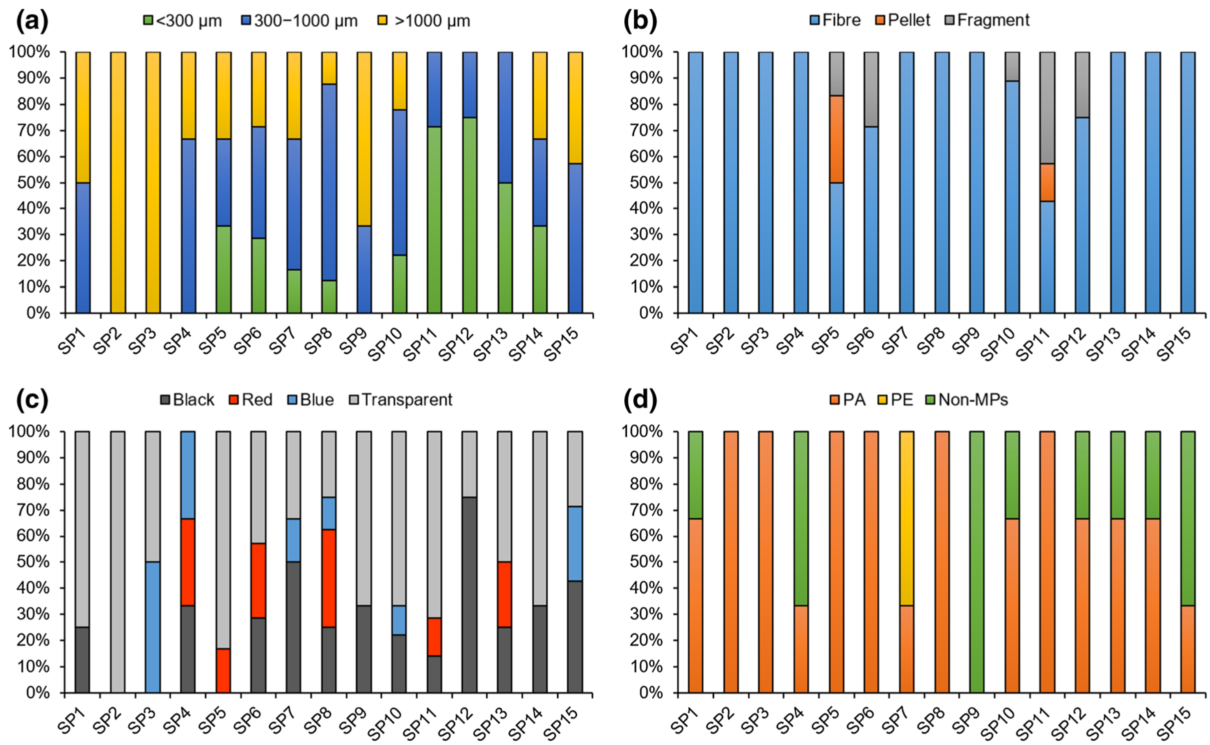


Fig. 4 Abundance and distribution of MPs in surface water of Klang River estuary, classified by **a** size, **b** shape, **c** colour and **d** polymer composition

MPs were categorised into < 300 μm, 300–1000 μm, and > 1000 μm classes with occurrence of 26%, 46% and 28%, respectively (Fig. 4a). The size of MPs observed ranged from 66.91 to 4133.33 μm across all sampling points in the Klang River estuary. Most MPs in surface water ranged in size from 300 to 1000 μm. The MPs were physically similar to plankton or prey in the environment and were, thus, potentially ingested by other organisms (Pegado et al., 2018).

MPs in surface water were classified as fibre, pellet, and fragment. The most common particles were fibres (85%) followed by fragments (11%) and pellets (4%) (Fig. 4b). The fibre particles may have originated from conventional activities such as fishing (Di and Wang, 2018; Wang et al., 2018; Yuan et al., 2019) and laundry (Napper and Thompson, 2016; De Falco et al., 2019). This indicates that the jetty, where fishing and boating activities such as vessel repair, maintenance, and cleaning mainly took place, influenced the abundance of MPs (Do Sul et al., 2014; Farias et al., 2018). This included discarded fishing gear (e.g. nets) exposed to various environmental conditions being fragmented and degraded. Similarly, frequent anthropogenic activities in the Klang River estuary, such as fishing and commercial and industrial activity, may contribute to the abundance of fibre.

In contrast, the presence of fishing activity did not influence the abundance of MPs along beaches of southeast India (Karthik et al., 2018). Proximity to the river mouth and improper plastic management are indicated as the main factors leading to MP pollution. In addition, fibre can be introduced to the environment in residential areas through the clothes washing process. A previous study revealed that microfibre is released into the environment during the washing of synthetic textiles (De Falco et al., 2019). Due to the mechanical and chemical forces during the washing process, synthetic fibres from textiles are shed and released into the environment (Hernandez et al., 2017). Fragments and pellets potentially originate from primary MPs widely used in personal care products and cosmetics (Cheung and Fok, 2016) which have been observed to contribute to MP pollution of the marine environment. Based on a previous study, wastewater treatment plants in Malaysia are expected to release 0.199 trillion of MPs, consisting of personal care and cosmetic products, into the country's marine environment (Praveena et al., 2018).

The colours of MPs were categorised as transparent, black, and coloured (red and blue) (Fig. 4c). Transparent MPs were the most abundant (50%) followed by black (28.4%), and coloured MPs (red, 12.2%; blue, 9.5%). Similarly, transparent MPs were the most abundant in estuarine water (Zhao et al., 2014; Anderson et al., 2018), sediment (Firdaus et al., 2020) and organisms (Li et al., 2018; Waite et al., 2018; Arias et al., 2019). Transparent MPs originate or are derived from plastic packaging (Wen et al., 2018), fishing lines (Di and Wang, 2018), and clothes (Wang et al., 2017). However, MPs can be overestimated and misidentified during microscopic observation due to the uncertainty of the period of exposure and conditions in the environment. Generally, MPs exposed for uncertain periods to environmental conditions, i.e. solar radiation, physical abrasion, and biological processes (Cole et al., 2011; Andrady, 2017), tend to undergo physical changes over time (Fahrenfeld et al., 2019). The physical changes include damage to the surface and discolouration of MPs. For instance, Zhang et al. (2016) found linear fractures, grooves, and mechanical pits in the surface morphology of MPs which were attributed to the windy conditions and mechanical erosion during transportation in remote lakes. Therefore, to reduce overestimation and misidentification of MPs during observation, infrared microscopy is needed, with a combination of visual identification, to verify the chemical and polymer composition (He et al., 2018).

This study identified 30 particles (71%) as MPs, while 12 particles were non-MPs. Any particles with FTIR spectra less than 70% were considered non-MPs (Yan et al., 2019). PA was the most frequently detected with 28 particles (67%) followed by PE with two particles (5%) (Fig. 4d). The ATR-FTIR spectra of PA and PE are shown in Fig. 5. The PA FTIR spectra indicate N–H, CH_2 (asymmetrical stretching) and CH_2 (symmetrical stretching) functional groups at 3265 cm^{-1}, 2916 cm^{-1}, and 2846 cm^{-1}, respectively (Razak et al., 2018). The adsorption bands at 1630 cm^{-1} and 1529 cm^{-1} are associated with C=O and C–N vibrations, respectively. The subsequent peaks at 1026 cm^{-1} indicate skeletal aliphatic C–H rocking vibration (Zarshenas et al., 2015). Consequently, the PE spectra show stretching vibrations of the $–CH_2$ groups at 2912 cm^{-1} and 2845 cm^{-1}. Visible adsorption bands were identified at 1470 cm^{-1} which represent C–C bonds (Razak et al., 2020). The adsorption bands at 1003 cm^{-1} and 710 cm^{-1} were assigned to C–H rocking and C–H out-of-plane deformation, respectively (Jung et al., 2018).

Approximately 28 particles of PA and two particles of PE were confirmed by ATR-FTIR. The MP extraction method of Masura et al. (2015) was used and optimised in this study to ascertain common MPs such as PE, PP, PVC, and PS. Although optimum percentage recovery was achieved during MP extraction, PP, PVC, and PS were absent in this study. Common MPs such as PE (0.97 g cm^{-3}) and PP (0.94 g cm^{-3}) are relatively less dense and are found in abundance floating on the surface water or higher in the water column, while denser MPs like PS

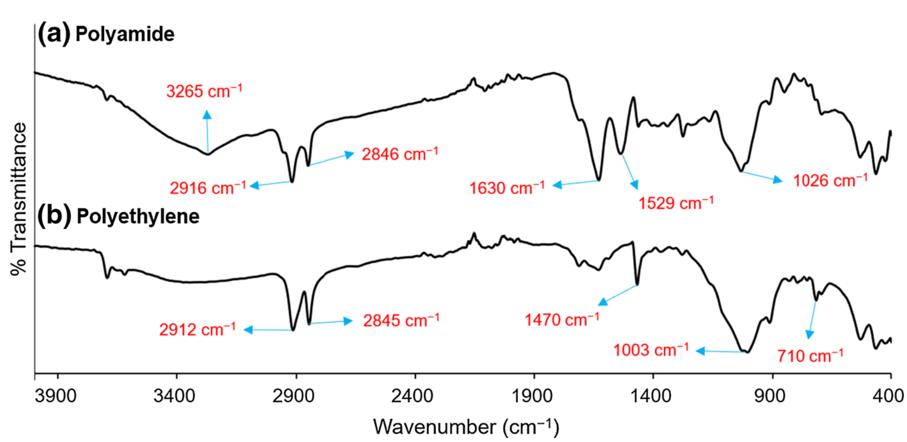


Fig. 5 FTIR spectra of polymer composition: **a** polyamide and **b** polyethylene

(1.05 g cm^{-3}), PA (1.15 g cm^{-3}) and PVC (1.38 g cm^{-3}) sink to the sediment (Cole et al., 2011; Lusher et al., 2013; Wang et al., 2018). This suggests that the types of MPs detected in this study may have been influenced by the density, buoyancy, and physical characteristics (e.g. size and shape). PA was the predominant synthetic polymer accounting for the highest proportion (26.2%) in the urbanised Pearl River estuary water (Yan et al., 2019), compared to 67% in the present study. PA is regarded as a high-density MP, and its occurrence and accumulation have been documented in sediment (Wessel et al., 2016; Scopetani et al., 2019; Egessa et al., 2020) and soil (Zhou et al., 2019). However, the findings of this study contradict the assumption that high-density MPs sink to the sediment, while low-density MPs float in the water column (Pegado et al., 2018). Despite having a higher density than freshwater or seawater, these denser MPs (i.e. PS, PA, and PVC) can float in the water column and be exposed to pelagic organisms. For instance, previous studies have shown denser MPs predominantly occurring in surface water in a lake (Wang et al., 2018), an estuary (Wu et al., 2019; Han et al., 2020), seawater (Zhang et al., 2017; Mai et al., 2018), and a river (Alam et al., 2019).

In this study, PA and PE were observed in locations in the Klang River estuary which are heavily influenced by wastes from fishing and maritime activities and industrial and residential areas. To identify the sources of MPs in the environment, the specific practical uses of polymer materials in modern society need to be determined. For instance, PA is the common type of polymer used extensively in fishing gear (e.g. fishing nets, lines, and ropes) and the clothing industry (Andrady, 2017; Liu et al., 2019). PP, PE, and PS are common plastics used predominantly in the packaging industry (e.g. plastic bottles), textile industry (Claessens et al., 2011; Di and Wang, 2018) and cosmetic products (Cole et al., 2011). These polymer materials have been found in various ecosystems, which may be related to the occurrence of PA and PE in the surface water of the Klang River estuary. In addition, the absence of PVC and PS in this study may be due to the density of the MPs, which eventually sink to the sediment from the water column (Andrady, 2017). Owing to small size (i.e. high surface area), MPs tend to adsorb contaminants and be subject to biofouling which influence buoyancy (Cole et al., 2011; Tang et al., 2018). Therefore, the reason for PP not being found in this study may be biofouling. This is supported by Firdaus et al. (2020) who documented the occurrence of PP and LDPE in sediment due to biofouling. In addition, environmental conditions, such as UV radiation, wave action, and turbulence, could influence the occurrence of MPs in the environment (Andrady, 2017). Ultimately, the factors that influence the availability and occurrence of MPs on the water surface are still poorly understood.

Conclusions

The results of this study indicate widespread occurrence of MPs in the surface water adjacent to urbanised areas of the Klang River estuary, Selangor, Malaysia. The abundance of MPs was higher in sampling points in relatively close proximity to residential, industrial, jetty, and port areas, which implies the contribution of anthropogenic activities and urbanised areas to MP pollution of the surface water of the Klang River estuary. The MPs were commonly fibres and were in the size range of 300–1000 μm. Transparent was the dominant colour observed, while PA was the major polymer composition of the MPs in all surface water samples. However, there was no significant correlation between physicochemical properties and the abundance of MPs in the surface water of the Klang River estuary. The high abundance of fibre and PA in this study may be attributed to anthropogenic activity such as fishing and transport. The mangrove area in the Klang River estuary provides crucial protection for the ecosystem and habitats for aquatic organisms. However, constant exposure of the estuary ecosystem to MPs raises ecological and health concerns. The preliminary occurrence of MPs in urbanised areas can be used for monitoring and comparative studies in the future. Further extensive studies on the occurrence of MPs in water, sediments, biota, and the atmosphere are crucial for comprehensive assessment of MPs sources, pathways, and fate in the environment.

Acknowledgement This project has been carried out under project Long Term Research Grant Scheme (LRGS/1/2016/UTM/01/1/5) provided by Ministry of Higher Education, Malaysia. Zaki M. R. M. would also like to acknowledge Wee, S.Y., Ismail, N.A.H., Nasir, H.M., postgraduate students, and laboratory staffs from Faculty of Forestry and Environment, Institute of Advanced Technology and Institute of Bioscience who directly or indirectly helped throughout the project.

Authors' contributions The study was designed by MRMZ, XYP, A.HZ, MRR, and AZA. The experiments were planned and performed by MRMZ, XYP, and AHZ. The modelling was performed by MRMZ, XYP, and MRR. The manuscript was written by MRMZ, XYP., AHZ, MRR, and AZA. All authors read and approved the final manuscript.

Funding This project has been carried out under project Long Term Research Grant Scheme (LRGS/1/2016/UTM/01/1/5) provided by Ministry of Higher Education, Malaysia.

Availability of data and material The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interests The authors declare that they have no competing interests.

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