Assessment of Microplastics and Potentially Toxic Elements in Surface Sediments of the River Kelvin, Central Scotland, United Kingdom

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Abstract

Contamination of the environment by microplastics (MPs), polymer particles of <5 mm in diameter, is an emerging concern globally due to their ubiquitous nature, interactions with pollutants, and adverse effects on aquatic organisms. The majority of studies have focused on marine environments, with freshwater systems only recently attracting attention. The current study investigated the presence, abundance, and distribution of MPs and potentially toxic elements (PTEs) in sediments of the River Kelvin, Scotland, UK. Sediment samples were collected from eight sampling points along the river and were extracted by density separation with NaCl solution. Extracted microplastics were characterized for shape and colour, and the polymer types were determined through attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy. Pollution status and ecological risks were assessed for both the microplastics and PTEs. Abundance of MPs generally increased from the most upstream location (Queenzieburn, 50.0 ± 17.3 particles/kg) to the most downstream sampling point (Kelvingrove Museum, 244 ± 19.2 particles/kg). Fibres were most abundant at all sampling locations, with red, blue, and black being the predominant colours found. Larger polymer fragments were identified as polypropylene and polyethylene. Concentrations of Cr, Cu, Ni, Pb and Zn exceeded Scottish background soil values at some locations. Principal component and Pearson's correlation analyses suggest that As, Cr, Pb and Zn emanated from the same anthropogenic sources. Potential ecological risk assessment indicates that Cd presents a moderate risk to organisms at one location. This study constitutes the first co-investigation of MPs and PTEs in a river system in Scotland.

Keywords: Microplastics; potentially toxic elements (PTE); freshwater sediments; the River Kelvin; risk assessment

Introduction

Plastic pollution is a major global environmental concern. Global production of plastic has increased from 1.5 million tons in the 1950s to 390 million tons in 2021 (Plastic Europe 2022). Plastic products have numerous applications, including food packaging, building and construction materials, electrical appliances, automotive components, medical devices, as well as household and sporting equipment (Bouwman et al. 2018; Plastic Europe 2020). This widespread use has led to an increase in the amount of plastic wastes ending up in the environment (Sayed et al. 2021).

Microplastics (MPs) are particles <5 mm in size and can be classified as primary or secondary microplastics based on their origin (Thompson et al. 2004). Primary MPs are deliberately manufactured in small sizes and can be released to the environment through spillage of plastic pellets during manufacturing or bulk transportation of such pellets, and through their use in household, medical and personal care products (Mehra et al. 2021). The presence of exfoliating microbeads is an instance of the use of MPs in personal care and cosmetic products. Secondary MPs originate from the degradation of large plastic items through chemical, physical and biological processes (Xia et al. 2021). Both primary and secondary microplastic particles enter rivers through surface run-off (from both rural and urban areas) and point sources such as wastewater treatment plants (WWTP) (Golwala et al. 2021; Knight et al. 2020; Su et al. 2019).

Once released into the environment, MPs are persistent because their degradation rates are slow (Yukioka et al. 2020). They pose risks to ecosystems, as they can be mistakenly ingested by organisms, due to their small size. They may therefore accumulate in higher organisms in the food chain (Karlsson et al. 2017). Ingested MPs can cause blockage and damage to the gastrointestinal tract of organisms. They also have toxicological and endocrine-disrupting effects. These arise as a result of additives incorporated during manufacture and chemical substances adsorbed on the

surface of the particles (Xu et al. 2020). Considerable research has demonstrated the ability of MPs to accumulate persistent organic pollutants such as polycyclic aromatic hydrocarbons and polychlorinated biphenyls (Fahrenfeld et al. 2019). More recent studies have also indicated that MPs can take up potentially toxic elements (PTE) from the surrounding environment (Ashton et al. 2010; Holmes et al. 2012; Turner and Holmes, 2015). Metals on the surface of MP can later desorb, or dissolve in the gastrointestinal fluids of organisms following ingestion (Song et al. 2017). Thus, MPs have the potential to act as vectors for PTE in natural systems.

Rivers are the predominant channel for the transport of microplastics to the oceans (Rochman, 2018). It has been estimated that between 0.41 and 4 million tons of plastic debris (comprising both MP and larger items) are transported to the ocean by rivers annually across the globe (Schmidt et al. 2017). Much of the MP's burden in rivers arises from urban areas due to their high population density and the large volume of waste generated (Su et al. 2020). Studies have been carried out on a number of river systems worldwide, including the Danube (Lechner et al. 2014), Seine (Gasperi et al. 2014), and Rhine (Mani et al. 2015; Mani and Burkhardt-Holm 2020). Most studies to date have focused on large rivers in areas of high population density (Jiang et al. 2022; Morgado et al. 2022; Shi et al. 2022), while small-scale rivers are less-often considered (though examples do exist e.g. Campanale et al. 2020; He et al. 2020; Kabir et al. 2021). Small-scale rivers are important freshwater systems, often rich in biological resources and therefore vulnerable to MP pollution. They are also the prominent conduits responsible for causing MP pollution in marine environments (Kabir et al. 2021). River sediments are a repository of microplastics and other contaminants such as potentially toxic elements (PTEs) in freshwater environments (Eo et al. 2021).

Levels of microplastics in river sediments have been reported to be several orders of magnitude greater than in the water column (Eo et al. 2019). Owing to the slow degradation rate of MP, they

accumulate in sediments (Li, Busquets, and Campos 2020). This accumulation further increases the ecological risk (Yin 2023). Although studies of MP in river sediments have been carried out on large rivers around the world (Mani et al. 2019), there is still a dearth of information on MPs in sediments of small-scale rivers (Kabir et al. 2022). Additionally, there are few studies that have addressed the subject of microplastic concentrations along a broad urban-rural gradient (Campbell et al. 2017; Chen et al. 2021; Dikareva and Simon 2019; Islam et al. 2023; Lahens et al. 2018; Matjasic et al. 2023). Analyses of this type are particularly critical, as their examination could reveal potential sources and delivery pathways of microplastic pollution.

The River Kelvin is a tributary of the River Clyde in west central Scotland, UK. It rises in the Kilsyth hills and flows 21 miles (33.5 km) approximately southwest to its confluence with the Clyde (Gazetteer Scotland 2022). The river initially passes through rural areas with livestock farming, then through residential suburbs, and finally through urban Glasgow. The City of Glasgow was formerly a hub for heavy industries (steel making, shipbuilding and chemicals manufacturing) and urban soils in the area contain elevated levels of PTEs (Davidson et al. 2006). A previous study (Blair et al. 2019) found 161 – 432 MPs kg^{-1} dry sediment at one specific site on the River Kelvin close to the River Clyde. However, the concentrations of PTEs in the sediment, and those adsorbed by the MPs were not investigated. The current study was aimed at the simultaneous investigation of the abundance and composition of MPs, as well as the levels and distribution of PTEs, in surface sediments taken from along the length of the River Kelvin, and examination of any relationships between the MPs and the PTEs. This is essential to a comprehensive understanding of the MPs' pollution and sources into the river

Materials and Methods

Study site and sample collection

The River Kelvin (latitude 55° 52' 12" – 55° 57'43" N and longitude 4° 5'14" – 4° 17'27" W) (Fig. 1) is a tributary of the River Clyde in West Central Scotland, UK. Its source lies in the central valley, in the neighbourhood of Kilsyth, and it flows 21 miles (33.5 km) westward and then southward, before entering the River Clyde at Partick, a Western suburb of Glasgow (Gazetteer 2022). The catchment of the River Kelvin comprises tributaries such as the Glazert, Luggie, Kilsyth, and Allander Waters, as well as a number of smaller streams (Moore et al., 2017). Historically, this river system has been vital to the human communities living within the catchment. Weirs and dams were built along the length of the river in order to provide power to mills, chemical works, shipbuilders, and paper and flint manufacturing companies. These various activities relied on the River Kelvin as a source of water, as well as for the disposal of their wastewaters.

Water from the River Kelvin was used to feed the summit level of the Forth and Clyde Canal. Its construction involved diverting some of the headwaters of the river into Townhead Reservoir, near Banton, east of Kilsyth (Hume, 2017). At Partick, the River Kelvin was used as water space for ship building and ship repairs from the 1840s to the 1960s. Today, the river passes through rural areas, with towns and villages in the vicinity, then through residential suburban, and finally through urban Glasgow and botanical gardens (SEPA, 2007).

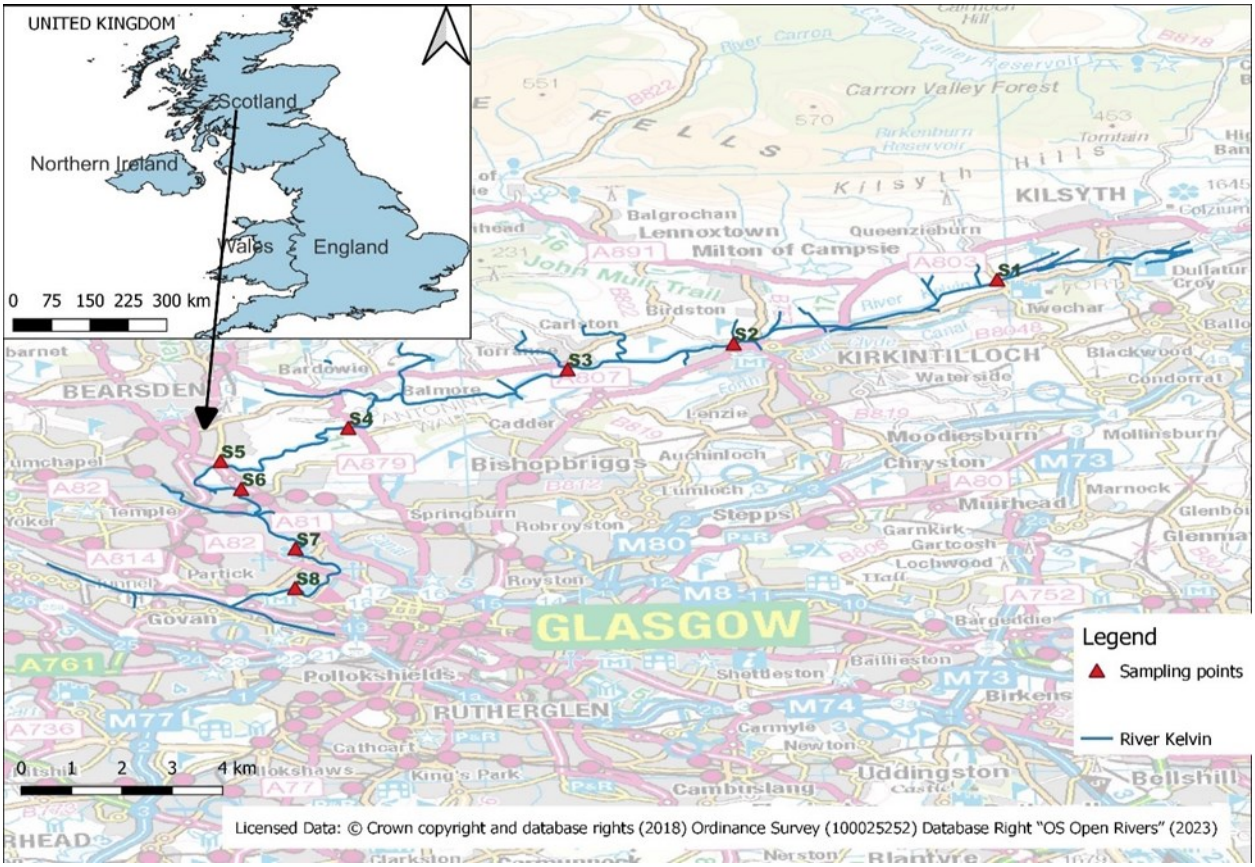

Fig.1 Map of the study location showing sampling sites on the River Kelvin, Scotland

Sediment samples were collected in September 2022 from eight sites along the length of the River Kelvin (Fig. 1). The sampling sites were designated S1 – S8 from upstream to downstream of the river (see supporting information Table S1). Sampling was performed as a composite from 3 points at each sampling site. The sediments were taken from a depth of 0 – 5 cm, using a stainless-steel trowel, and the samples were carefully transferred into wide-mouthed glass containers and transported to the laboratory for further processing.

Sample processing and microplastic extraction

Sediment samples were processed and MP was extracted using the density separation method (Thompson et al. 2004; Zhao et al. 2018) with slight modifications. Briefly, 30 g of each air-dried and previously sieved (through a 5 mm mesh-size sieve) sample were weighed in triplicates into 1-Litre glass beakers, and 200 mL of saturated sodium chloride (NaCl, density 1.2g/cm^3, Fischer Scientific, UK) solution was added and stirred thoroughly with a clean glass rod for 2 min. The added NaCl solution was prepared with deionized water and filtered through a 0.45 µm membrane filter to remove potential plastic particles and other impurities before use. After 2 h of settlement, the suspension was carefully decanted into another glass beaker, to which 5 mL of hydrogen peroxide (30 % H_2O_2) (Fischer Scientific, Loughborough, UK) was added for the digestion of organic matter. The solution was left in a fume hood for 24 h to allow sedimentation to occur, then the supernatant was filtered through a nitrocellulose membrane filter (Fisher Scientific 125 mm diameter, pore size 0.45 µm) under vacuum. In order to reduce sample loss due to possible adhesion of MPs on the beaker walls, each glass beaker was rinsed with a copious amount of pre-filtered milli-Q water, and the content was passed through the same membrane filter as the sample. The filter was carefully removed using clean tweezers and placed in a clean petri dish to dry before microscopic analyses.

Microscopic analysis

To determine the quantity, shape, colour, and size of the microplastics recovered, the dried filter papers were observed and photographed (at 40 to 100x magnification) using a digital handheld microscope (Bysameye, China). Abundances were recorded as items/kg dry sediment, the various microplastics shapes present were identified, and the size of the recovered microplastics was determined using imaging software (ImageJ, v.1.53k).

Microplastics polymer identification

To ascertain the type of polymers, large MP fragments were analysed using attenuated total reflectance - Fourier transform infrared spectrometry (ATR-FTIR). The FTIR analysis was done using a Nicolet™ iS5 infrared spectrometre (Thermo Fisher Scientific, UK) at a spectral range of 400 – 4000 cm^{-1}. Each spectrum involved 16 scans at a resolution of 4 cm^{-1}. The identified spectra were processed by OMNIC™ polymer spectra software. The resulting spectra were thereafter compared with the software's reference databases for identification of the plastic polymers (Hummel polymer, Aldrich polymers, etc.).

Determination of potentially toxic elements (PTEs)

The method of Ashton et al. (2010) and Dobaradaran et al. (2018) with slight modifications was used for the extraction of PTE from large MP fragments. Each fragment was placed in a 10 mL glass beaker and 2.5 mL of 20% aqua regia solution was added, and the beaker was shaken for 12 h at 150 rpm and 25 °C.

For the determination of pseudo total (aqua regia soluble) PTEs concentrations in sediments, 1.0 g of each dried sample was weighed in triplicate into digestion vessels, 20 mL of aqua regia (3:1, HCl: HNO_3) (Fischer Scientific, Loughborough, UK) was added, and the mixture was left overnight for any vigorous reaction to subside. The mixtures were subjected to microwave-assisted digestion using a MARSXpress (CEM, Buckingham, UK) digestion system. After digestion, the vessels were allowed to cool and the sample digests were filtered into 100 mL volumetric flasks. The digestion vessels were rinsed two more times and the mixture was filtered into the flask; the digest was then made up to 100 mL with deionized water.

Digests were analyzed for arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), manganese (Mn), nickel (Ni), vanadium (V), and zinc (Zn) by inductively coupled plasma-mass spectrometry (ICP-MS) using an Agilent 7700x system (Agilent, Cheshire, UK). The instrument was calibrated using multielement standards and the internal standards were: ^{45}Sc, ^{72}Ge, ^{115}In, and ^{209}Bi. The operating conditions of the instrument were: RF power 1550 W; nebulizer gas flow rate 0.85 L/min; plasma gas flow rate 5.0 L/min; auxiliary gas flow rate 0.9 L/min, and cooling gas flow rate 15 L/min. The certified reference material LGC 6187 (River sediment – Extractable metals) was analyzed to determine the accuracy and precision of the method. The average recoveries were between 87 and 123% (Table 1). Triplicate digestion and analyses were carried out on each sediment and certified reference material (LGC 6187) and the precision (RSD) was less than 10% in all cases.

Table 1 Percentage recovery of potentially toxic elements in certified reference materials (LGC 6187) (River sediment – Extractable metals)

PTEs	Certified value	Measured value	Recovery (%)
As	25.7	24	93
Cd	3.0	2.7	90
Cr	97	84	87
Cu	85	83.6	98
Pb	62.9	77.2	123
Mn	1310	1240	95
Ni	32	34.7	108
V	40.8	38.3	94
Zn	425.1	439	103

Risk assessment of microplastics and PTEs in sediments of the River Kelvin

The MP pollution risk assessment model developed by Kabir et al. (2021) was used to determine the contamination factor (CF) and pollution load index (PLI) of the microplastics in the river. The

values of CF and PLI indices are categorized from low to very high contamination (see Supporting Information Table S2).

To assess the PLI for each site and river:

$$CF_i = C_i / C_o \quad \text{(i)}$$

$$PLI = \sqrt{CF_i} \quad \text{(ii)}$$

$$PLI_{river} = \sqrt[n]{PL_1 \times PL_2 \times PL_3 \times PL_n} \quad \text{(iii)}$$

where i represents a sampling point on the river, n represents the total number of sampling points, C_i represents the abundance of MP particles at sampling point i, C_o is the minimum mean background abundance value taken as the least amount of MP particles determined at any of the sampling points. PLI_i is the pollution load index at the sampling point i and PLI river is the MP pollution load index for the entire river. Sites are polluted when PLI > 1.

The level of PTEs pollution in the sediments of the River Kelvin was assessed by determining the CF, PLI, geoaccumulation index (*Igeo*), and potential ecological risk index (PERI) (see supporting information, Table S3).

Contamination control

Plastic materials were avoided throughout the study to prevent possible contamination. Sampling was conducted using a stainless-steel trowel, which was rinsed with pre-filtered deionized water between sites to prevent cross-contamination. Prior to use, glassware was soaked in 5% nitric acid for 24 h and rinsed with copious amount of pre-filtered deionized water. To prevent airborne MP contamination, all samples and working tools were covered with aluminum foil when not in use. Extracted MPs were kept in clean petri dishes, and procedures were carried out under a fume hood. Windows were closed during analysis to minimize airflow. A 100% cotton laboratory coat and

nitrile gloves were worn throughout the laboratory processing. All solutions used were filtered with a nitrocellulose membrane filter (Fisher Scientific 125 mm diameter, pore size 0.45 µm) prior to the extraction of MPs to remove impurities and other particles. Blanks were processed at the same time as samples and no microplastics were found in the blanks.

Statistical analyses

Statistical analyses were performed using the statistical package for social sciences (SPSS, V 26). Correlation and principal components analyses (PCA) were used to check for significant relationships among PTEs in the sediment samples. The various statistical methods were performed with a 95% confidence interval (significance level at $p < 0.05$). Factor analysis based on PCA was used to ascertain sources of contamination (natural or anthropogenic).

Results and Discussion

Abundance of microplastics in the sediments of the River Kelvin

Some MP particles were found in sediments from all sampling locations (Fig. 2). A total of 1260 items/kg of dry-weight sediment was found from all sampling points. The mean MP abundance values (mean ± SD) ranged from 50.0 ± 17.3 to 244 ± 19 items/kg of dry sediment (average 147 items/kg) (Fig. 2). Highest abundances occurred at sampling point S6, which is located at Dawsholm Park, and at point S8 (located in an urban park behind the Kelvingrove Museum) with 211 ± 107 and 244 ± 19 items/kg, respectively. Point S6 is close to an outfall from the surface drainage system discharging large amount of stormwater, while point S8 is heavily impacted by recreational activities around the Museum. A sharp rise in the abundance of MPs was observed at point S4 (near a landfill), when compared to the three upstream locations (S1 to S3). The results agree with a previous study which showed that landfill is a source for MP-derived debris to enter into aquatic environments (Sulistyowati et al. 2022). In contrast, the lowest abundance of 50.0

± 17.3 items/kg was recorded at point S1, which is located at Queenzieburn in the upstream area. At this site the river runs through arable land and, although it is close to a village, the human population density is lower than at the other sampling locations. The high MP abundances at sites S6 and S8 could also be attributed to population density, tourism, and anthropogenic activities such as trade and commerce, industries, and urban development. Relationship between MP abundance and population density has been established by a few studies, notably Townsend et al. (2019), on the sediments of wetlands in the Greater Melbourne Region, Victoria, Australia. Meanwhile, site S7 had a lower abundance compared to S6; this reduction may be due to the swift flow of the river observed at that location when sampling, which may have limited deposition of MPs on sediments.

In general, the upstream section of the River Kelvin presented lower MP abundances of 50, 88, and 100 items/kg for sites S1, S2, and S3, respectively. Likely sources of MP in these upstream regions are diffuse (non-point) inputs such as run-off from agriculture. Plastics could also be introduced by direct inputs from precipitation and wind (Duis and Coors 2016). Furthermore, MP pollution in the area could come from general sources like the wearing of tyres, as well as from road pavements containing polymer-modified bitumen. The distribution of MP in sediments across sampling location follows the order: S1 (3.98%), S2 (7.08%), S3 (7.96%), S4 (14.6%), S5 (15.0%), S6 (16.8%), S7 (15.1%), and S8 (19.4%). The highest PLI value (2.21) was obtained for sampling site S8, followed by S6 (2.06), S5 and S7 (both 1.94), S4 (1.91), S3 (1.41), S2 (1.33) and S1 (1.0). The overall PLI along the length of River Kelvin is 2.8, indicating that the river may be classified under the risk category I of the pollution index, which implies slight pollution by the MP. The higher PLI values obtained for S8, S7, S6, and S5 could be due to the collection of surface water in the urban drainage network and discharge into the river nearby those sampling points, as

well as recreational activities in some areas. In contrast, the lower PLI at S1 could be a result of lesser anthropogenic activities around the area.

Table 2 shows the MP abundance in this study in comparison with other studies published within the last decade (2015 – 2023) in freshwater sediments around the globe. The MP abundances reported in this study are lower than those reported in freshwater sediments in Yan River, China (209 – 687 items/kg) (Zhao et al. 2023), Ayaragi River, Japan (24 – 608 items/kg) (Kabir et al. 2022), and Fu River, China (212 – 1049 items/kg) (Zhou et al. 2021). The values are however higher than those reported for Yushan River, China (30 -70 items/kg) (Niu et al. 2021), Qin River, China (0 – 97 items/kg) (Zhang et al. 2020), Yonfeng River, China (5 – 72 items/kg) (Rao et al. 2020), and Asa River, Japan (8 – 182 items/kg) (Kabir et al. 2022). Overall, the foregoing indicates that the MPs abundance for River Kelvin, determined in the current study, are within the range of values reported for most freshwater sediments around the world.

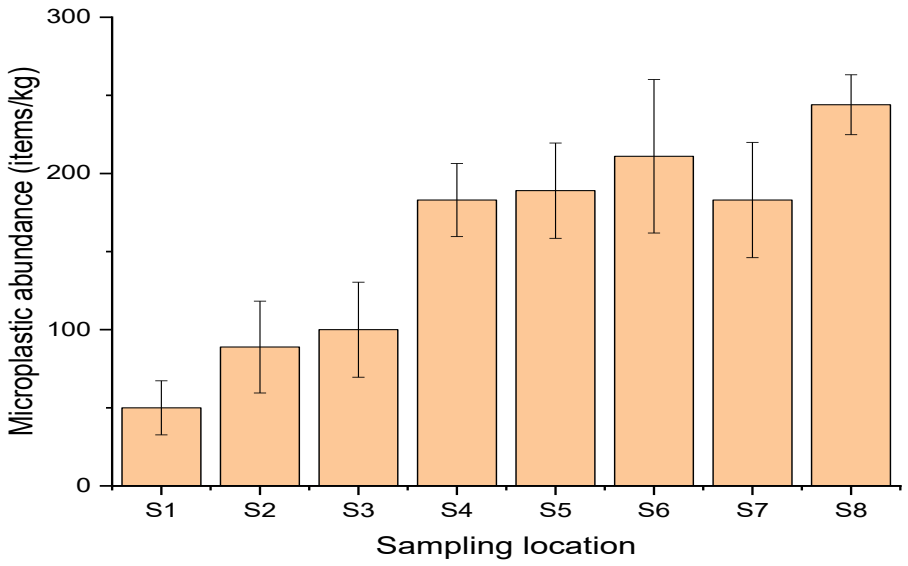

Fig.2 Abundance of MP in the sediments of River Kelvin, Glasgow, United Kingdom

Table 2 Abundance of microplastics (MPs) in sediments of the River Kelvin, UK, compared to values in sediments of some rivers across the globe (items/kg dry weight sediment)

Study areas	Abundance (items/kg)	Size (mm)	Shape	Polymer	Reference
River Kelvin, UK	50.0 -244	0.05 -5	Fibres, fragments	PE, PP	This study
Rhine River, Germany	228 – 3760	0.063 – 0.6	Fragments, sphere	PE, PP, PS	Klein et al. (2015)
Main River, Germany	786 – 1370	0.063 – 0.6	Fragments and Spheres	PE, PP, PS	Klein et al. (2015)
Taihu lake, China	11.0 – 235	0.1 – 1.0	Fibres	Cellophane	Su et al. (2016)
Otawa River, Canada	220	NA	Fibres	NA	Vemaire et al. (2017)
Beijiang River, China	178 ± 69 – 544 ± 107	NA	NA	PE, PP	Wang et al. (2017)
River Tame, UK	165	< 5	Fibres, fragment, films	PE, PVC	Tibbettes et al. (2018)
Pearl River, China	80.0 – 960.0	0.05 – 0.5	Fibres	PE, PP	Lin et al. (2018)
Antua River, Portugal	100 – 629 18 – 514	NA	Fragments	PP, PE	Rodrigues et al. (2018a), (2018b)
Wei River, China	360 – 1320	0.075 – 0.5	Fibres	PE, PVC, PS	Ding et al. (2019)
River Kelvin, UK	161 - 432	0.011 – 2.88	Fibres, fragment	NA	Blair et al. (2019)
Yonfeng River, China	5.00 – 72.0	< 1	Films	PE, PP	Rao et al. (2020)
Qin River, China	0.000 – 97.0	1 – 5	Fibres and sheets	PE, PP	Zhang et al. (2020)

Amazon River, Brazil	417 – 8180 0 – 5730	0.063 – 5 0.063 – 1	Fibres	NA	Gerolin et al. (2020)
Brisbane River, Australia	10.0 – 520	< 3	Fibres, fragments, and films	PA, PE, PP, PET	He et al. (2020)
Tisza River, Central Europe	3180 ± 1970	NA	Fibres, pellets, fragments	NA	Kiss et al. (2021)
Fuhe River, China	212 – 1050	0.1 – 5	Fragment, fibres, film, pellet	PS, polyamide, PP, PE	Zhou et al. (2021)
Yushan Urban River, China	30.0 – 70.0	NA	Fibres, films	PE, PP, PET	Niu et al. (2021)
Ayaragi River, Japan	24.0 – 608	1 – 5	Fragments, films	PE, PP, PVA	Kabir et al. (2022)
Asa River, Japan	8.00 – 182	1 – 5	Fragments, films	PE, PS, PVC	Kabir et al. (2022)
Yan River, China	209 – 687	0.5 – 1	Fibres, foam, films, fragments	PS, PE, PP, PET	Zhao et al. (2023)

NA – not available; PA – Poly-amide; PE – Polyethylene; PET – Polyethylene terephthalate; PP – Polypropylene; PS – Polystyrene; PVC – Polyvinyl chloride; UK – United Kingdom

Shape of microplastics in the sediments of the River Kelvin

Common shapes of MPs in the environment include fibre/line, fragment, foam, pellet, sphere and film (Akhbarizadeth et al. 2018). Various of these shapes have been detected in freshwater sediments, with fibres being the most commonly reported. Fig. 3 shows the typical morphological characteristics of MPs observed in the sediments of the River Kelvin. In this study, it is noteworthy that almost all of the MPs recovered from the sediments were fibres; only two fragments were found. Fibre microplastics emanate from various sources, the chief being the widespread use of synthetic (polymer) as clothing materials. Thus, washing and laundry of clothes result in the release of huge amounts of MP fibres (Browne et al. 2011), which are released from household discharge through washing activities. A pipe discharging detergent-laden water was seen near the sampling point at S6, Dawsholm Park. Since there is no wastewater treatment plant in the vicinity, and untreated domestic waste is not typically discharged directly to water bodies in Scotland, this cannot originate from laundry activities in households. A possible explanation is the presence of a car wash in the vicinity. Such facilities can release copious amounts of soapy water to the urban drainage network and may have contributed to the level of fibre MPs generally observed in this area. Fibres also commonly enter aquatic environments through fishing activities. However, there are no commercial fisheries in the vicinity of the River Kelvin. Fragments normally emanate from the breakdown of large plastic items. Fragment MPs were found only at S4 and S8 in this study. The fragment at S4 is consistent with the presence of a landfill where breakdown of discarded plastic items could be occurring. The fragments at S8 may originate from the discard and subsequent breakdown of plastic bottles or food packaging items, since this is an urban park frequented by members of the public, with a major tourist attraction (Kelvingrove Museum) in the vicinity. The relative abundance of fibres and fragments in the current study is in agreement with

the report by Alam et al. (2019), who found 91% (fibres) and 9% (fragments) as the composition of MPs from the sediment of Ciwalengke River, Indonesia. Peng et al. (2017) also reported fibre to be the most prevalent shape (93%) in the sediment of Changjiang Estuary, China. In other studies, in the Nakdong River and Wen-Rui Tang River, fragments were found to be the main form of microplastics (Wang et al. 2018; Eo et al. 2019). In contrast, the dominant shape of MPs recorded in the sediment of the Brisbane River, Australia, was film, followed by fragments and fibres (He et al. 2020). Fragment particles mostly originate from the degradation of larger plastic particles exposed to strain, fatigue and UV light (Wen et al. 2018; Xiong et al. 2018; Zhang et al. 2016). Measuring the particle shapes is important to understanding the impact of MP on biota, as coarse fragments are more injurious to the digestive system of aquatic species if ingested (Dusaucy et al. 2021).

Size, colours and polymer characteristics of microplastics from the River Kelvin sediments

The size of MPs greatly affects their migration in the aquatic environment and their potential for ingestion by organisms. In this study, frequently observed MP sizes ranged between 50 and 2000 µm, with the majority having sizes below 1000 µm (Fig.3). This result is consistent with previous studies (for example, Blaskovic et al. 2017 and Lots et al. 2017), which showed that MPs with particle size < 1 mm were more abundant in freshwater sediments.

Colour is an important characteristic that determines the impact of MPs in aquatic systems. This is because coloured MPs are easily mistaken for food by some aquatic organisms (He et al. 2020; Rodrigues et al. 2018). The MP particles from the sediment of the River Kelvin are categorized as shown in Fig. 3. The figure confirms the presence of brightly coloured MPs, as well as black and white ones. Coloured MPs could originate from a number of plastic consumer products with long service lives. Brightly coloured MP fibres found in the environment (such as the ones shown

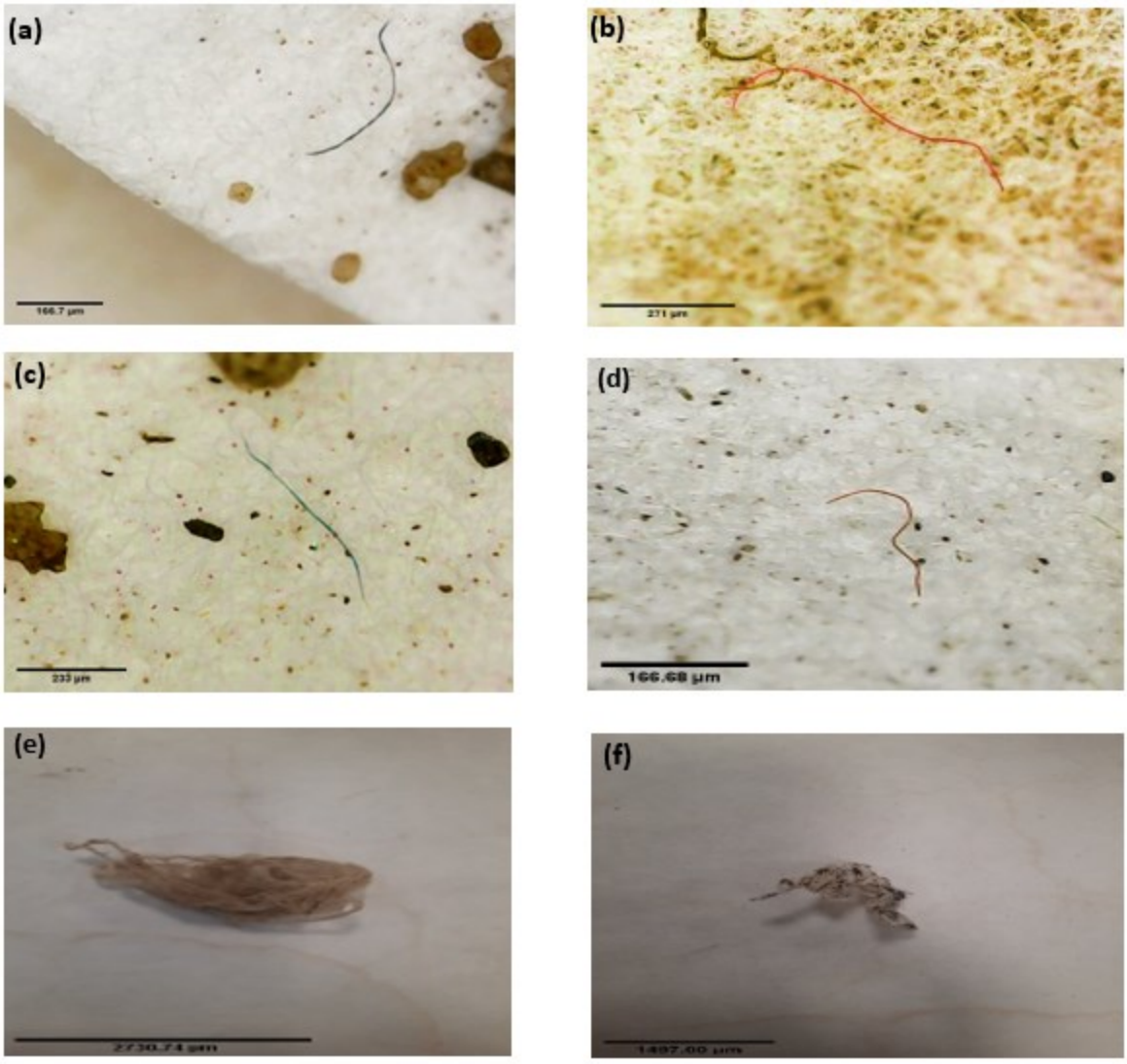

Fig.3 Images of fibres (a – d) and fragments (e – f) microplastics recovered from the sediments of the River Kelvin, Scotland, United Kingdom

in Fig. 3) are usually associated with the manufacture and use of coloured garments (Eo et al. 2019; Browne et al. 2011; Wen et al. 2018). Furthermore, coloured MP could be one of the factors for the ingestion of MP by biota due to their resemblance to their prey (Wright et al. 2013).

The polymer nature of MPs from the sediments was investigated by ATR-FTIR. Of the two fragments found in the sediments of the River Kelvin, one was made of polyethylene (PE) and the

other of polypropylene (PP) (Fig.4). Both PE and PP polymers are used in the production of a great variety of consumer items: PE is used in food packaging materials, trays, toys, and disposable bags, whilst PP is used in food packaging, bottle caps, snacks and sweet wrappers (Plastic Europe 2020). Together, PE and PP are the most commonly used plastics due to their low cost and ease of processing (Phuong et al. 2018). It is therefore not surprising that these polymers are found in the sediments. Furthermore, the presence of PP polymers may not be unconnected with the recent global COVID-19 pandemic, where a huge number of face masks (made of PP fabric) were used

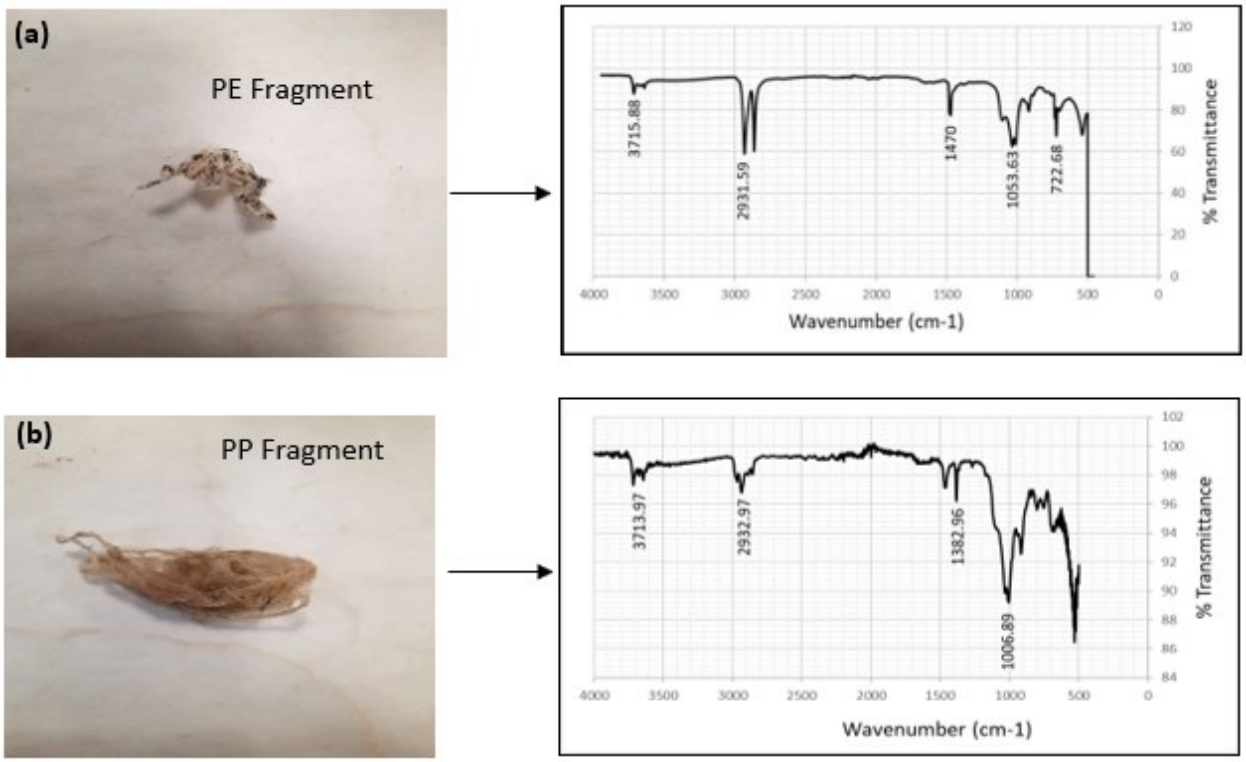

Fig.4 Representative polyethylene (PE) **(a)** and polypropylene (PP) **(b)** microplastics recovered from the River Kelvin sediments and their corresponding FTIR spectra.

and inappropriately disposed of in the open environment. A recent study by Idowu et al. (2023) demonstrated that these face masks degrade under ambient outdoor conditions to release PP MPs into the environment. The prevalence of PE MPs in the environment has also been attributed to their durability and high resistance to photo-degradation (Zhu et al. 2020).

Potentially toxic elements (PTE) associated with microplastics and sediments of the River Kelvin

The concentration of PTE was determined for both the sediments and the MPs recovered from them. The mean concentrations of As, Cd, Cr, Cu, Mn, Ni, V, and Zn in the sediments of the River Kelvin are compared with the world rivers background and average background Scottish soil values (Table 3). The average concentrations (mg/kg) of As, Cd, Cr, Cu, Mn, Ni, V, and Zn were 9.67, 0.275, 33.4, 27.6, 1400, 17.3, 63.9, 56.1, and 127 mg/kg, respectively. These average concentrations are in the decreasing order of Mn > Zn > Pb > V > Cr > Cu > Ni > As > Cd. The concentrations of all PTEs varied along the length of the river (Table 3). The highest concentrations of PTEs were generally recorded in the mid-sections of the river at S5, followed by S4 or S6. These high values may be attributed to anthropogenic influence e.g. location S5 is beside a busy main road and motor trader. Manganese was the element found at highest concentration in the sediments (564 to 2570 mg/kg), while Cd concentrations were the lowest (0.022 to 0.501 mg/kg).

Levels of Mn in the sediments are higher than the USEPA limit value (460 mg/kg) in both upstream and downstream sections of the river but did not markedly exceed the World Rivers background of 1680 mg/kg. The small enhancement observed could be due to the dissolution of geologic minerals in the upstream area and anthropogenic activities (e.g. run-off from landfill) in the midstream). Copper and Zn measured in the sediments at some of the sampling points exceeded the Scottish soil background value for these elements. The elevated Cu and Zn concentrations

observed at location S6 might be due to coal-tar residues which may still exist in the vicinity of Dawsholm woodland. Concentrations of As measured in this study at S5 and S6 were higher than the USEPA value (9.8 mg/kg), but was lower in the sediments from the other locations The high values at S5 and S6 may be attributed to anthropogenic activities. Arsenic was used to treat wood at timber and shipyards in the 1960s, and chromated copper arsenate was applied to timber used for fencing as recently as 2006. The Cd values were lower than the Scottish soil background concentration (0.16 mg/kg) at a few locations, but higher at locations S1 – S5. The slightly elevated levels at these locations are probably due to a combination of anthropogenic sources e.g. vehicular emissions, fertilizer use, and municipal runoff. The Cr and Ni values were lower than the Scottish background soil concentration (44.7 mg/kg), except at location S5 where they were slightly exceeded, perhaps due to the presence of a garage nearby. Overall, concentrations of Cd, Cr, Cu, Ni, Pb and Zn at some locations were higher than background Scottish soil values, as expected since the river runs through areas impacted by human activity.

The average concentrations of PTEs in the sediment of the River Kelvin were compared with PTEs concentrations reported by related studies (see supporting information Table S4). The concentrations of As, Cd, Cu, and Ni, in the sediments of the River Kelvin, were lower than those reported for sediment in Aries River, Romania (Moldovan et al. 2022) and sediments of Rivers in Poland (Sojka and Jaskula. 2022).

The mean concentrations (µg/g) of PTEs adsorbed on PP were (As – 2.85 ± 2.0, Cd – 27.8 ± 1.0, Cr – 8.05 ± 0.5, Cu – 154 ± 3.5, Mn – 5420 ± 35, Ni – 327 ± 0.5, Pb – 25.2 ± 2.1, V – 31.6 ± 1.8, Zn – 30.6 ± 2.1), while those adsorbed on PE particle were (As - 1.65 ± 0.0, Cd – 0.0 ± 0.0, Cr – 0.37 ± 0.0, Cu – 0.0 ± 0.0, Mn – 2.07 ± 0.1, Ni – 0.0 ± 0.0, Pb – 0.0 ± 0.0, V – 0.34 ± 0.0, Zn – 0.0 ± 0.0) . The mean levels of Pb, Mn, Ni, Cu, and Cd in sediments were greater than that of MP

particles at some locations, while the levels of Cd, Cu, Mn, and Ni in the recovered polypropylene MP at location S4 were higher than those obtained in the sediments of the same location. The presence of PTE on MP may be attributed to the additives used in the original plastics, as well as adsorption of PTE from the sediment. The higher concentration of PTE on the surfaces of MP than in the sediment at location S4, confirms the hypothesis that MP can accumulate and act as carriers and vectors of PTE in the environment. Values of PTEs obtained were similar to those reported by Sarkar et al. (2021), who worked on the occurrence of microplastics as heavy metal vectors in the sediment of wetland systems in India (see supporting information Table S5).

Assessment of PTE pollution in the sediments of the River Kelvin

The geoaccumulation index (I_{geo}) values of the nine PTEs in the sediments of the River Kelvin are in decreasing order of Pb (0.77) > Zn (0.1) > Mn (-0.03) > V (-0.70) > As (-1.21) > Cd (-1.25) > Cr (-2.13) > Cu (-2.51), and Ni (-2.68) (see also supplementary information, Fig. S1). The negative I_{geo} values ($I_{geo} < 0$) for Cu, Cr, Ni, and V at all sampling locations suggest that the sediments are not polluted by these PTEs and that the values obtained are mostly from lithogenic contributions. The I_{geo} values for As, Mn, and Zn were also less than zero for all sampling sites, except S4 and S5 which were above zero ($0 \leq I_{geo} < 1$). This indicates that the sediments were moderately contaminated by As, Mn, and Zn and had minimal anthropogenic input in this study area. The mean I_{geo} values of Pb and Zn in the sediments are between 0 and 1 (low pollution). However, at individual sampling points, Pb reaches 0 to 1 (low pollution) and 1 to 2 (moderate pollution) levels. The mean I_{geo} value of Pb (2 to 3) is much higher than other PTEs, showing that the River Kelvin is most polluted with Pb. Possible sources of Pb pollution include Pb batteries and historical vehicular emissions. Higher accumulation of Pb and Zn in river sediments have also been reported by other similar studies (Dai et al. 2018; Fang et al. 2019) and was mostly attributed to

Table 3 Concentrations of PTEs (mean ± SD, mg/kg dry samples) in sediments of the River Kelvin

Sampling site	As	Cd	Cr	Cu	Mn	Ni	Pb	V	Zn
S1	3.89 ± 0.0	0.411 ± 0.0	26.4 ± 0.8	22.2 ± 1.8	2470 ± 4.2	22.2 ± 2.6	24.3 ± 0.6	65.1 ± 1.1	116 ± 3.1
S2	9.01 ± 0.2	0.446 ± 0.0	16.8 ± 0.7	14.6 ±0.6	1680 ± 0.7	22.9 ± 0.1	22.7 ± 0.6	75.4 ±1.3	141 ±0.7
S3	4.62 ± 0.0	0.220 ± 0.0	21.6 ± 1.1	18.0 ± 0.2	1540 ± 1.0	14.4 ± 0.3	30.5 ± 0.3	53.8 ± 0.6	115 ± 0.5
S4	7.31 ± 0.3	0.362 ± 0.0	38.0 ± 0.6	24.1 ± 0.5	2260 ± 1.3	17.5 ± 0.5	55.9 ± 0.0	57.2 ± 0.4	134 ± 0.6
S5	29.7 ± 0.0	0.501 ± 0.0	69.8 ± 0.3	43.8 ± 0.0	1180 ± 2.2	29.4 ± 0.1	154 ± 0.1	57.5 ± 0.1	158 ± 0.7
S6	14.7 ± 0.6	0.153 ± 0.0	34.5 ± 0.2	54.1 ± 0.6	775 ± 0.2	15.3 ± 0.1	115 ± 0.2	50.8 ± 0.9	127 ± 0.2
S7	5.30 ± 0.0	0.081 ± 0.0	32.6 ± 0.7	27.6 ± 0.1	564 ± 0.3	8.21 ± 0.3	44.3 ± 0.2	45.5 ± 0.0	102 ± 0.4
S8	8.92 ± 0.1	0.022 ± 0.0	27.4 ± 0.3	16.3 ± 0.2	772 ± 0.6	8.50 ± 0.0	65.2 ± 0.7	43.7 ± 0.1	120 ± 0.1
Scottish Soil[a] Background	-	0.16	44.7	9.4	-	26.5	31.8	-	53.8
World Rivers Background[b]	36.3	1.55	130	75.9	1680	74.5	61.1	129	208
UCC[c]	1.5	0.098	85	25	-	44	17	-	71
USEPA[d]	9.8	0.99	43.4	31.6	460	22.7	35.8	-	121

NB: As, Arsenic; Cd, Cadmium; Cr, Chromium; Cu, Copper; Mn, Manganese; Ni, Nickel; Pb, Lead, V, Vanadium; Zn, Zinc. (n = 3)

[a] MLURI (2003)

[b] Viers et al. (2009).

[c] Taylor and McLennan (1985)

[d] USEPA (1999)

anthropogenic sources.

The highest values of CF for Mn (2.91), Pb (7.69), Zn (2.23), As (2.28) and Cd (1.67) were recorded at S5, indicating moderate to high contamination (see supplementary information, Table S6). In contrast, lower CF values were obtained for Pb (1.14), As (0.354), Cd (0.270), Cr (0.304), Mn (0.60) at S2, S1, S7, S8, and S7, respectively. The pollution load index (PLI) helps to assess the overall pollution status of soil or sediments. In the present study, PLI values are < 1 in all sampling sites, except S5 which has a PLI value > 1 (see supplementary information, Fig. S2). This indicates that only sampling point S5 is polluted with PTEs. The highest PLI observed in S5 (midstream area) suggests greater anthropogenic influence than the other points. Overall, the PLI determined for the entire River is 1.15 (PLI > 1). This result implies that River Kelvin is slightly polluted with the PTE.

The potential ecological risk factor (E^i_r) values for the nine PTEs follows the order: Cd (22.2) > Pb (15.9) > As (8.01) > Cr (5.85) > V (1.87) > Zn (1.78) > Mn (1.65) > Cu (1.45), and Ni (1.27) (supplementary information, Table S7). The As, Cr, Cu, Mn, Ni, V, and Zn E^i_r values are below 40 at all sampling locations which suggest a low ecological risk from these PTEs to aquatic organisms. However, E^i_r value for Cd was greater than 40 at S5 indicating that Cd poses a moderate ecological risk to organisms in the river sediment, consistent with the PLI index.

Identification of sources of PTEs in sediments of the River Kelvin

Principal component analysis (PCA) is a widely used multivariate analysis to evaluate the potential similarities, sources (anthropogenic or natural), and characteristics of PTEs in sediments (Uddin et al. 2021). In the present study, PCA was used to evaluate possible PTE sources, using the varimax rotation method. The PCA extracts two components with eigenvalue > 1 and explains 91.92% of the total variance. It shows that 58.38% of the total variance was explained by the first

component (PC1) and 33.55% was explained by the second component (PC2) (supplementary information, Table S8 and Fig.S3). The elements Cd, Cu, Mn, Ni, and V had greater positive loading in PC1, indicating that these PTEs could be similar in behaviour and have emanated at least in part from geogenic or natural sources in the sediments of the River Kelvin. This is supported by the geo-accumulation index where Cu, Ni, and V have $I_{geo} < 0$. The PC1 can thus be considered to represent mixed sources of lithogenic and anthropogenic activities. In contrast, As, Cr, Pb and Zn had the greatest loadings on PC2, which implies that these PTEs could be from human sources in the sediments of the River Kelvin. This is also supported by the high CF value of As (2.28) and Pb (7.69) in the study site. These PTEs mostly have inputs from the local point and non-point sources such as surface water runoff, and atmospheric deposition.

Pearson correlation was applied to examine relationships between PTEs (supplementary information, Table S9) and gave positive significant correlations for Cu, Ni, V ($p < 0.05$) and Zn ($p < 0.01$), implying that Cd had a common source with Cu, Ni, V and Zn, and that these elements have similar geochemical characteristics. Significant positive correlations were also observed between Cr and Pb, and between As and Zn ($p < 0.01$), indicating that these pairs of elements may have originated from similar sources.

Conclusions

This study has investigated the abundance and composition of MPs, as well as the distribution of PTEs, in sediments of the River Kelvin, Scotland, UK. Microplastics were found at all sampling locations. The overall abundance of MP observed in the sediment was 1260 items/kg, increasing from the upstream to the downstream section of the river. Fibres were the most abundant microplastic particles in the sediments. The colours of the recovered MPs were mostly red, blue, and black. Polyethene and polypropylene fragments were found in the sediments. Concentration

of the PTEs in the sediments followed the order of Mn > Zn > Pb > V > Cr > Cu > Ni > As > Cd. Potential ecological risk assessment indicates that there is a moderate risk of exposure to Cd, while other PTEs have no ecological risks. PCA analysis suggests that As, Cr, Pb, and Zn originated from anthropogenic sources in the study area. Being the first study to investigate MP pollution along a significant length of the River Kelvin, this work is of value for progressive monitoring of MPs and to improve understanding of their role in transporting PTE in the river system. The study also contributes to knowledge on the presence of microplastics in small-scale rivers flowing along rural-urban transects. Whereas most previous studies globally have focused on major/large rivers, the small rivers are the first receptacle of MPs from terrestrial environments and, thus, play an important role in contributing to marine MPs pollution.

The main limitation of the study was the scarcity of large-size MPs, which could have facilitated a more comprehensive investigation of the relationship between PTEs associated with MPs and those in the sediments. The application of further analytical techniques – for example micro-Raman spectroscopy to determine the polymer types for fibres – would also have been desirable. These limitations notwithstanding, the findings from this study serve as a useful baseline for future investigation of MPs and their relationship with PTEs in the sediment of the River Kelvin

Declaration of competing interest

The authors declare no competing financial interest

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