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Research article

Microplastics in freshwater environment: the first evaluation in sediment of the Vaal River, South Africa

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HIGHLIGHTS

- MPs were 100% prevalent in sediment of the Vaal River.
- Fragments, small-sized, and coloured MPs were most prevalent.
- Seven types of polymers were identified.
- Wastewater and anthropogenic activities were identified as major sources of MP pollution.

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ABSTRACT

Microplastic pollution has become an environmental concern worldwide. In this study, the occurrence, abundance, and composition of microplastics (MPs) in sediment of the Vaal River, South Africa were assessed. Twenty-five sediment samples were collected from the Vaal River using a Van Veen grab sampler, samples underwent digestion, density separation, and filtration prior to physical and chemical analysis. Following the extraction, potential MPs were visually identified under a Nikon stereomicroscope, aided by chemical characterization using Raman spectroscopy. The results revealed 100% prevalence in sediment samples, with an average abundance of 463.28 ± 284.08 particles/kg_dw. Small-sized MPs of 2 mm and less were the most abundant, representing more than 82% of the total particles. Fragments and coloured MPs were the most dominant compared to other shapes and transparent particles, accounting for 63% and 60%, respectively. Microplastics were identified as polyethylene (PE) (both high and low density), polypropylene (PP), and polyethylene co-vinyl acetate (PEVA), polyester (PES), polyurethane foam (PU), and polyethylene/hexene-1-copolymer (PEH). These findings reveal elevated levels of MP contamination within the Vaal from secondary sources. Potential sources include wastewater effluent, anthropogenic activities, surface run-off from urban centres, inflow from tributaries, and recreational activities.

1. Introduction

Microplastics (MPs) are among some of the most deleterious emerging contaminants that humanity has to contend with following a boom in the plastics industry in the 20th century. The variety of plastics manufactured; their non-biodegradability; bioaccumulation; lack of recycling; and lack of data regarding their distribution have exacerbated the potential impact of these contaminants on the environment [1, 2].

MPs can be intentionally manufactured in the microscopic size (primary) or result from the fragmentation of larger plastics (secondary) [3, 4, 5, 6, 7]. They enter the aquatic environment via multiple pathways, including atmospheric fallout, effluents from wastewater treatment plants (WWTPs); as well as runoff from agricultural, recreational, industrial, and urban areas [8, 9, 10, 11, 12]. Due to their small size and resemblance to natural food items, MP particles are consumed by different aquatic species, in incidental or intentional ways [13, 14, 15, 16, 17].

In addition to their possible health risks, the potential hazard associated with MPs includes exposure to hazardous substances such as plastic additives and toxic chemicals that are absorbed from the ambient matrices, as well as pathogenic microorganisms colonizing the plastics into the aquatic food web [4, 18, 19, 20, 21, 22].

Studies have shown that ingestion of MPs by aquatic organisms can affect their feeding behaviour, reproduction, and growth; once ingested, MPs have the potential to be passed up through the food chain [19].

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Thus, MPs ingestion by aquatic organisms presents a potential exposure route for humans via seafood consumption.

Globally, MP research has primarily focused on marine system, but, the past few years have seen a shift in focus to studying MPs in freshwaters [9]. However, until recently, less than 4% of MPs-related studies have addressed MP pollution in freshwater systems [23]. In South Africa, there is a dearth of knowledge on MP occurrence in freshwater bodies, with much limited data on freshwater sediments [24]. Studying MPs in freshwater sediment is important to better understand their distribution, fate, and bioavailability to deposit feeders and benthic organisms. The aim of this study was to investigate the occurrence and abundance of MPs in sediment of the Vaal River and to identify potential sources.

2. Methodology

2.1. Study area and sample collection

The Vaal River is the main tributary to the Orange-Vaal River, South Africa's largest freshwater system. It is a major freshwater body in South Africa, providing water supply for agricultural and industrial activities, as well as drinking [25]. 98% of raw water used by Rand Water (the largest water utility in South Africa that supplies potable water to the Gauteng province and other areas in the country) is extracted from the Vaal River. Once purified, it supplies water to about 926 industries, 40 mines, and 13 municipalities. Essentially, the Vaal River services 11 million people in Gauteng, Mpumalanga, North West, and the Free State provinces, and contributes more than 50% to the GDP and at least 80% to the electricity supply [25, 26]. The Vaal is also a scenic host to a variety of recreational activities including fishing, boat cruises, and water sport [27].

Samples were collected using a 500 mL Van Veen grab sampler; a rope was tied to the hanger of the Van Veen and slowly lowered into the river bed from the side of the boat. A total of 25 samples were collected at 3 km intervals and at depths of 5–10 cm. At each sampling site, 3 replicates were scooped and mixed in a 2 L glass beaker using a stainless-steel spoon. A 325 mL representative sample was then placed in precleaned glass jars with lids lined with aluminum foil to prevent contamination. The jars were transferred to the laboratory in a cooler box and stored in the refrigerator at 4 °C until analysis. A map depicting the sampling locations is given in Figure 1.

2.2. Samples treatment and MPs extraction

2.2.1. Drying and digestion

Samples were first oven-dried at 40 °C for 10 days, until a constant mass was reached. Alkali digestion was then performed by adding 10% KOH to the dry sediment samples at a volume ratio of 1:3. KOH has been reported to have minimal damage to the MPs and is recommended for samples with high organic matter [28]. The mixture was stirred for a few minutes, covered with aluminum foil, and heated on a hot plate at 40°C for 24 h to achieve complete digestion before separation [25].

2.2.2. Density separation and filtration

Sodium Iodide (NaI) was used to extract MPs based on their density; 80 g powder was dissolved in 100 mL ultra-pure distilled water to prepare a saturated NaI solution (1.8 g cm^{-3}). This solution was then added to the digested sample in a volume ratio of 1:3, stirred for 5 min, and left for 24 h. Samples were then vacuum filtered through a GF/A filter paper Whatman®: 47 mm diameter, pore size: 1.6 μm. The filter papers were allowed to air-dry overnight and stored in glass petri dishes until analysis.

2.3. Morphological and chemical identification of microplastics

Following extraction, potential MP particles were visually identified according to their physical characteristics (shape, size, and color); aided by chemical characterization to identify polymer composition.

2.3.1. Physical identification

Visual criteria proposed in the literature were adopted to identify MPs in this study. This includes the absence of cells, organic structure and metallic luster, unnatural shapes (spherical and fragments), and homogeneous texture [29]. Furthermore, the "break test" was used to differentiate between plastic and non-plastic particles. Tweezers were used to

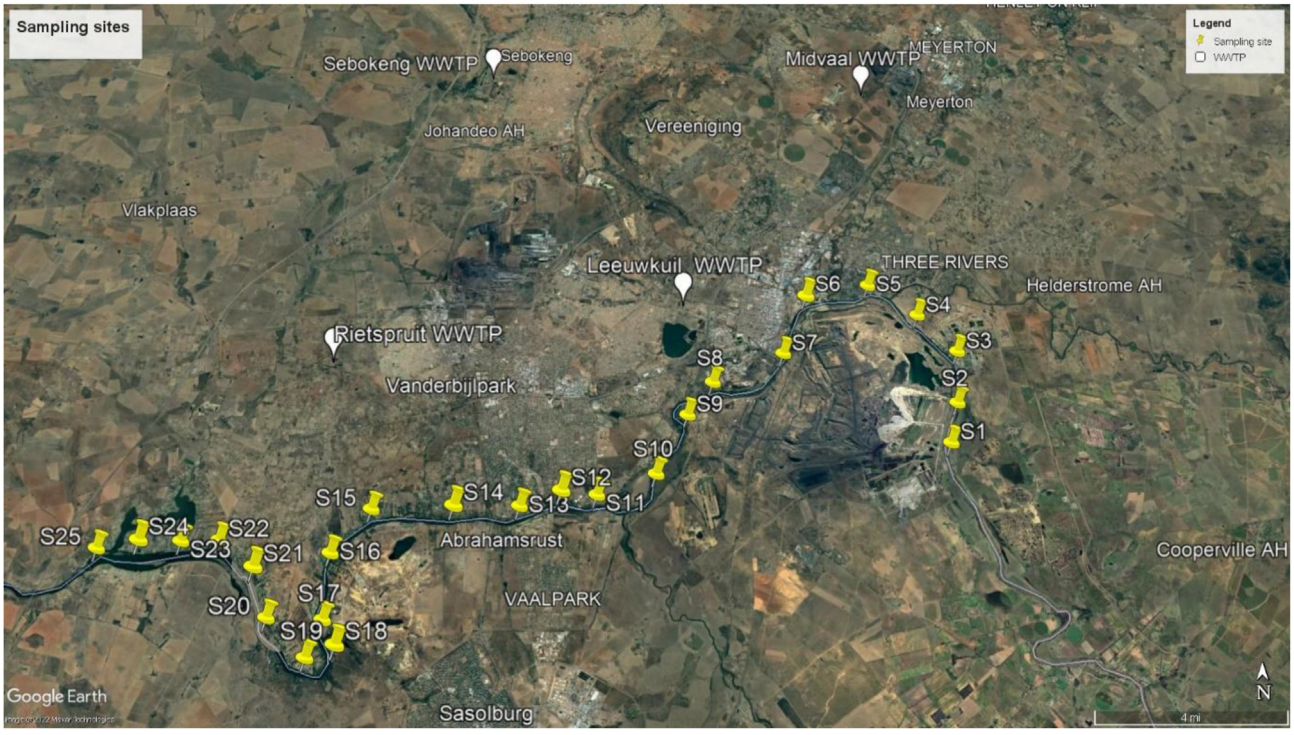

Figure 1. Map of sampling area (Google Earth, 2021).

Table 1. Microplastics number and abundance per sample.

Sample code	Dry mass (kg)	Number of MPs	Abundance (particles/kg_dw)
S1	0.25	68	270.00
S2	0.08	75	937.50
S3	0.13	115	884.62
S4	0.14	82	585.71
S5	0.12	83	691.67
S6	0.14	79	564.29
S7	0.09	47	522.22
S8	0.18	44	244.44
S9	0.11	76	690.91
S10	0.18	86	477.78
S11	0.37	28	75.68
S12	0.03	12	400.00
S13	0.05	4	80.00
S14	0.41	23	56.10
S15	0.27	8	29.63
S16	0.09	55	611.11
S17	0.14	98	700.00
S18	0.07	25	357.14
S19	0.08	19	237.50
S20	0.06	24	400.00
S21	0.34	28	82.35
S22	0.04	19	475.00
S23	0.06	44	733.33
S24	0.07	72	1028.57
S25	0.10	40	400.00

inspect the MPs; the particles that broke when probed were not considered MPs, as plastic particles are flexible and spring when pressed [30]. Potential MPs were identified under a Nikon stereomicroscope (Nikon MET SMZ745T, Japan). The particles were photographed with an imaging source camera (TIS) USB 3.0 and processed with NIS Elements-D imaging software Version 5.30 (Nikon Cooperation, Japan). In addition to identifying their shape and colour, the software was also used to count the number of particles as well as to measure particle size (with an upper size of 5 mm).

Scanning Electron Microscopy (SEM) analysis was carried out in order to examine the surface structures of MPs using TESCAN Vega. Selected MPs of different shapes were transferred onto the double-sided adhesive carbon tabs on aluminum stubs, gold-coated and subjected to variable pressure in secondary electron mode.

2.3.2. Chemical characterization

Raman spectroscopy (Horiba LabRAM HR) was used to examine the chemical composition of the potential MPs. The resulted spectra were compared with reference spectra in the SLOPP Library of microplastics and polymer databases of KnowItAll software (Bio-Rad Laboratories, Inc.) to identify polymer type.

2.4. Quality control measures

Sampling and sample processing were performed using metal and glass equipment and containers. Cotton lab coats and gloves were worn during sampling and sample processing, and all experiments were performed under a laminar-flow hood. The extracted MPs were stored in pre-cleaned covered petri dishes. Cross-contamination was controlled by blank control experiments. No microplastics were detected in the blank samples, which indicated low potential of contamination in the laboratory environment.

3. Results and discussion

3.1. Quantification of MPs

Microplastics were prevalent in all sediment samples, with abundances ranging from 29.12 to 1095.89 particles/kg_dw, and a mean abundance of 463.28 ± 284.08 particles/kg_dw (average ± std dev).

Table 2. Abundance of MPs in sediments of different freshwater bodies.

Study area	Freshwater system	Sampling apparatus	Abundance (particles/kg_dw)	Reference
Qinghai-Tibet, Tibet	Rivers	Spatula	41.52 ± 22.31	(Feng et al., 2021)
Auckland, New Zealand	Rivers	Bucket	80	(Dikareva and Simon, 2019)
Taihu Lake, China	Lake	Peterson grab	893.48 ± 245.74	(Zhang et al., 2021)
Wei River, China	River	Sludge sampler	360–1320	(Ding et al., 2019)
Lake Ziway, Ethiopia	Lake	Ekman grab	0.05–36.233	(Merga et al., 2020)
Oxbow Lake, Nigeria	Lake	Grab sampler	347 - 4031 and 507 - 7593	(Oni et al., 2020)
Bloukrans River, South Africa	River	N/A	160.1	(Nel et al., 2018)
Braamfontein Spruit, SA	River	N/A	166.8	(Dahms et al., 2020)
Vaal River, South Africa	**River**	**Van Veen grab**	**463.28 ± 284.08**	**This study**

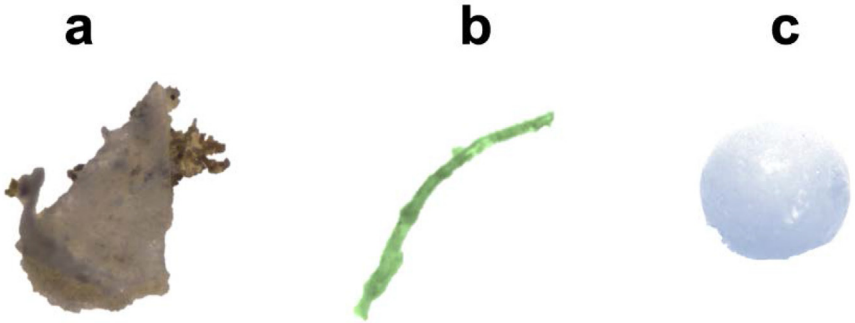

Figure 2. Microscope images of major MPs shapes: a) brown fragment, b) green fiber, and c) a transparent pellet.

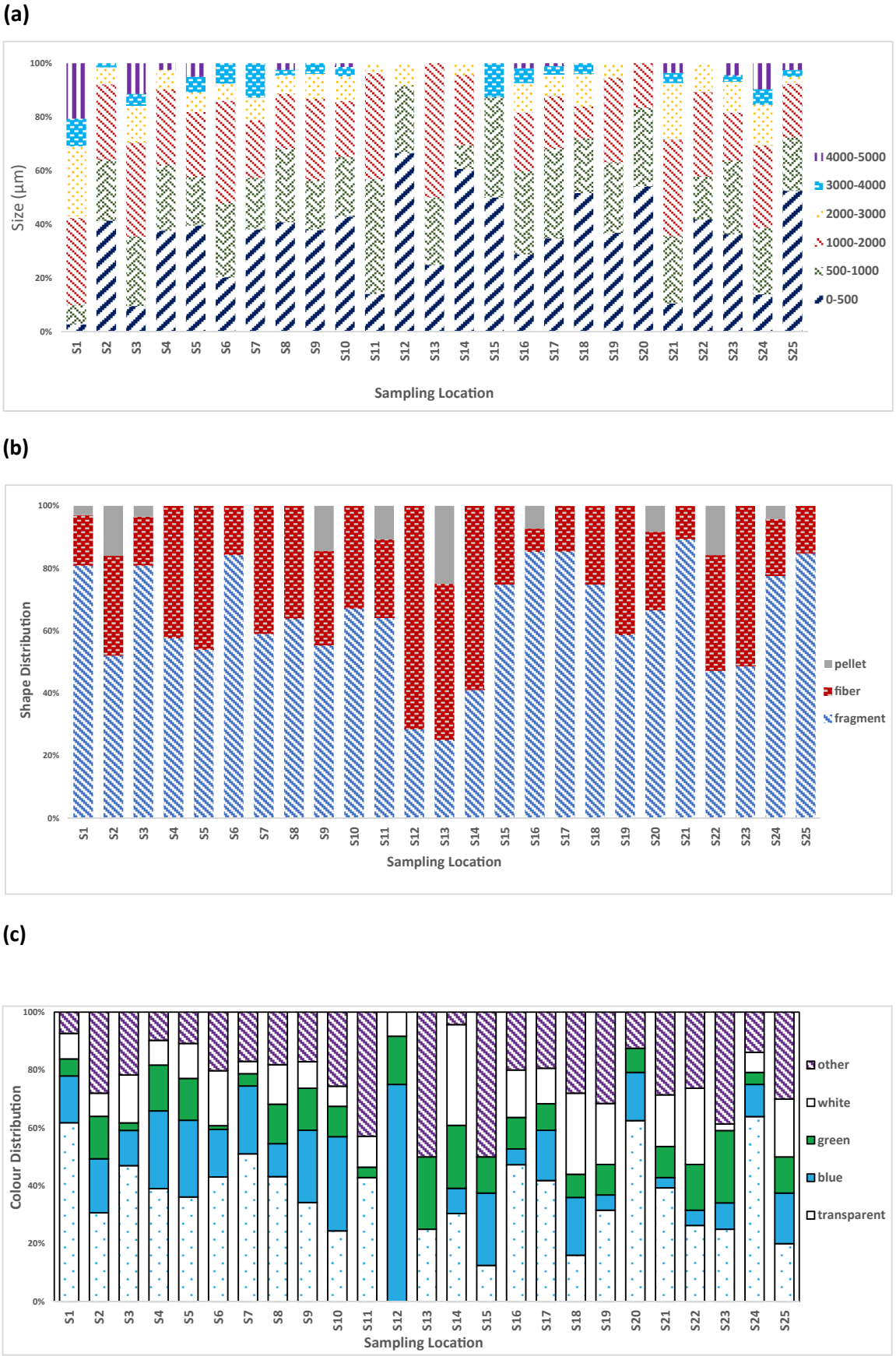

Figure 3. Distribution of (a) size, (b) shape, and (c) colour per sample.

Abundance was determined as particles per kg of dried weight of the sediments. The number and abundance of MPs per sample are given in Table 1.

The results above are an indication of MP pollution in the Vaal River. Potential sources in the area include water and wastewater treatment plants (e.g., Rand Water and Emfuleni Municipal wastewater treatment plant), industrial activities (e.g., Lethabo Power Station and ArcelorMittal), and waste generated from surrounding residential areas (e.g., Sedibeng and Metsimoholo). Urban centers are reported to be major sources of plastic pollution in several studies [31, 32, 33, 34]. Additionally, the Vaal is a scenic host to a variety of recreational activities including fishing, boat cruises and houseboats, and water sports (rafting, tubing, flyboarding, kayaking and river boarding). There are several resorts, restaurants and hotels along the river bank, examples within the sampling area are Emerald Resort, animal world, the Aquadome, and Eligwa boat club, to mention a few. Plastic litter discarded by tourists could therefore be a significant contribution to the high presence of fragments in the water body [27].

Also, some samples were collected from spots near the confluences of the Vaal with its tributaries. The major tributaries feeding into the Vaal are Klip River and the Leeuspruit. The Klip River flows from some densely populated areas such as Soweto and Lenasia [35]. Whereas, Leeuspruit drains Sasolburg, the home to Sasol which is well known as an integrated energy and chemical company. Tributaries are known to have significant input on the pollution of main rivers. Dahms et al. (2020) reported a steep increase in MP abundance at a confluence of the Braamfontein Spruit and Montgomery Spruit. Similarly, Zhang et al. (2015) observed that four tributaries of the Yangtze River had a significant contribution to its MPs abundance [36, 37].

3.1.1. Comparison with other freshwater studies worldwide

While some freshwater studies have revealed similar findings, others have reported significantly different concentrations of MPs (Table 2). For instance, a recent study reported a comparable pollution level in Taihu Lake in China, with a mean abundance of 893.48 ± 245.74 particles/kg_dw [38]. Similarly, Ding et al. (2019) reported comparable findings in Wei River, China; with abundance values ranging from 360 to 1320 particles/kg_dw. In both studies, the authors attributed the high abundance of MPs to discharges from industrial, agricultural, and other anthropogenic activities, which explains the similarity with the pollution status in the Vaal River. Globally, higher MP abundance in rivers is frequently reported in those within populous urban areas [39, 40], as exemplified by the Vaal River.

Examples of freshwater bodies that are reported to have much lower MP contamination include the Qinghai-Tibet Plateau River in Tibet and eighteen streams in Auckland, New Zealand [41, 42]. A varying abundance of 4–1347.5 particles/kg_dw in sediment samples from the Braamfontein Spruit in Johannesburg, South Africa was reported by Dahms et al. (2020).

3.2. Physical identification of MPs

MPs were visually identified under a Nikon stereomicroscope based on their (<5 mm), shape, and colour. Microscopic images of potential MPs of different shapes are shown in Figure 2.

Fragments were dominantly present in all samples (63%), followed by fibers (35%), and pellets (2%). The dominance of fragments and fibers is consistent with other freshwater studies [30, 41, 43, 44, 45, 46, 47]. The distribution of shapes in sediment samples is given in Figure 3 (b).

The high prevalence of fragments and fibers over pellets clearly indicates that MP pollution is of secondary sources. Fragments originate from the degradation of large plastic debris and are transported through effluent discharges. In the context of the Vaal River, fragments could be attributed to different anthropogenic activities around the Vaal, notably,

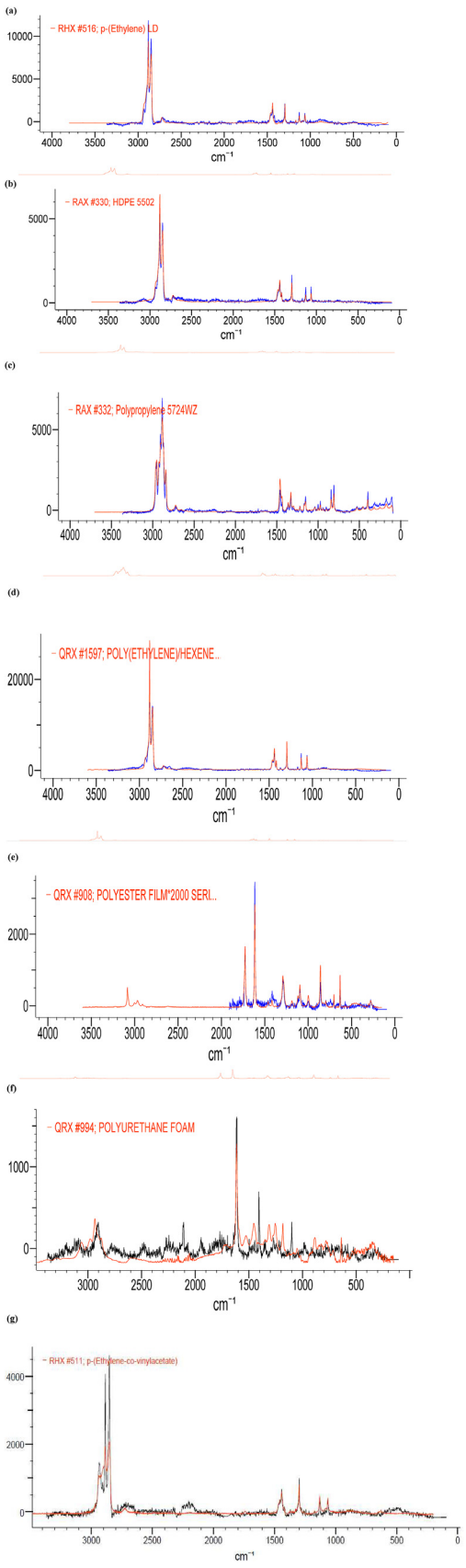

Figure 4. Raman spectra of (a) LDPE, (b) HDPE, (c) PP, (d) Polyethylene/Hexene-1 copolymer, (e) Polyester, and (f) Polyurethane, and (g) Polyethylene co-vinyl acetate.

the section of the river sampled is well known for a variety of recreational activities. Consequently, picnics, water sports, and waste generated from the hotels and resorts may contribute to MP pollution. Furthermore, pieces of plastics from fishing rods, nets, and boats are directly introduced to the river; this is evident by torn fishing nets and fishing lines encountered during sampling. Once in the river system, the relatively larger surface area of fragments facilitates the adsorption of other contaminants from the surrounding environment which enhances their sedimentation [48]. The dominance of fragments in sediment was reported in several studies, for example, in Qinghai-Tibet Plateau, Tibet; in Lake Taihu, China; and in Lake Ziway, Ethiopia, to mention a few [38, 42, 49].

Fibers are mostly attributed to wastewater treatment plants and domestic sewage around the Vaal [50, 51]. Machine washing of clothes discharges high quantities of MP fibers into domestic wastewater, a large proportion of which could pass through the filtration systems of WWTPs, mostly smaller size, and enter the river system [6, 51].

In terms of size, it was observed that smaller sizes less than 0.5 mm are the most abundant, which accounts for 31.75% of the total particles, followed by 23.84% of MPs in the range of 0.5–1 mm, 26.56% in the range of 1–2 mm, and 17.85% in the range of (2–5 mm). The distribution of these sizes is shown in Figure 3 (a). The dominance of small-sized MPs of <500 μm in sediment was reported in several studies [38, 52, 42, 45, 53]. This could be attributed to the large surface area of MPs, which facilitates bio-fouling and attachment of other contaminants, thus reducing the buoyancy and further enhancing the settling capability [48, 54].

Coloured MPs accounted for 60% as shown in Figure 3(c). A great variety of colorant agents such as pigments and dyes are widely used during plastic manufacturing, thus, a large number of coloured plastic wastes are produced with the consumption of these products. Transparent MPs represented a significant percentage of the total particles detected, accounting for 40%. However, it is worth noting that, bleaching processes may occur in the aquatic environment leading to some coloured plastics to become transparent [51].

3.3. Chemical identification

Post-visual identification, the composition of potential MPs was further examined by Raman spectroscopy. Seven polymers were confirmed (Figure 4), namely, high-density polyethylene (HDPE), low-density polyethylene (LDPE), polyethylene co-vinyl acetate (PEVA), polypropylene (PP), polyester (PES), polyurethane foam (PU), and polyethylene/hexene-1-copolymer (PEH).

Polyethylene (PE), polypropylene (PP), and polyethylene co-vinyl acetate (PEVA) were the most common polymers found among the MPs. Their prevalence is consistent with MPs from secondary sources as they are frequently used in single-use plastics, packaging, textiles, and containers, in addition to industrial, domestic, and agricultural applications [55, 56, 57, 58].

4. Conclusions

This study is the first attempt to investigate MPs presence and abundance in sediment of the Vaal River. Our findings revealed elevated levels of MP pollution in the river, with 100% prevalence. Fragments and fibres were more abundant compared to pellets, which indicates that MP pollution in the Vaal River is from secondary sources. Wastewater effluent and anthropogenic activities around the Vaal such as fishing, agriculture, tourism, and industrial activities were identified as potential sources.

The pollution of the Vaal River is of particular concern considering the economic value of the river; South Africa relies on the Vaal for multiple uses (drinking water, agriculture, industries and power generation). However, our findings provide a benchmark on which future studies can hinge; as well as evidence that are necessary in informing considerations for relevant policy and decision making.

Declarations

Author contribution statement

Dalia Saad: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Michelle Ndlovu; Gibbon Ramaremisa: Performed the experiments; Wrote the paper.

Hlanganani Tutu: Conceived and designed the experiments; Wrote the paper.

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Data availability statement

Data will be made available on request.

Declaration of interest's statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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