PAPER

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Microplastic occurrence in sediments of informal settlement in Durban, South Africa: assessing the ocean impact in the aftermath of floods

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The environmental impact of microplastics is increasingly being recognized, leading to their inclusion as contaminants of emerging concern. Consequently, it is essential to identify and monitor microplastics and their impact on the ecosystem. In this study, a ball miller was employed to process plastic waste, generating microplastics for the optimisation of separation methods. Microplastics of various sizes and shapes were produced and used to spike the sediments for optimisation of the density separation method. The recovered microplastics ranged from 0.74–5 mm, as confirmed by stereomicroscopy. In addition, the types of polymers present in microplastics were confirmed using Fourier Transform Infrared Spectroscopy-Attenuated Total Reflectance (FTIR-ATR). The developed analytical method was employed to study the occurrence of microplastics in river sediments passing through informal settlements after floods. Moreover, the evaluated data confirmed that informal settlements are a major source of microplastics found in the ocean after a flood. Microplastics in river and ocean sediments were predominantly composed of polypropylene, polyethylene, and polystyrene with a size range of 0.90–5 mm. To the best of our knowledge, this study is the first to report the impacts of informal settlements on the ocean during floods in Africa.

Environmental significance

This research highlights the pervasive issue of microplastic pollution in water systems, particularly in areas affected by floods where microplastic concentration can increase due to factors like runoff and sediment transport. Understanding the distribution and abundance of microplastics in these sediments is crucial for comprehending the extent of plastic pollution in aquatic environments and its potential impact on marine ecosystems. By examining the presence of microplastics in sediments post-flood events, this study provides valuable insights into the dynamics of plastic waste transport, accumulation, and the subsequent implications for water quality, biodiversity, and ecosystem health in coastal regions. This research aids in formulating effective strategies for mitigating microplastic pollution and safeguarding the marine environment against the detrimental effects of plastic contamination.

1 Introduction

Plastics exposed to the environment do not degrade rapidly; instead, they break into smaller particles and gradually undergo degradation over time, influenced by various environmental conditions.[1–3] Plastics entering the environment have recently become a significant environmental concern as they break down into nanoplastics and microplastics, classified as contaminants of emerging concern (CECs).[4–6] Microplastics can serve as effective environmental indicators, helping us understand whether rivers passing through informal settlements transport significant contaminant loads into the oceans.[7] Given the physiochemical properties of plastics, microplastics do not form during flooding, making them perfect indicators of years of river pollution by informal settlements. MPs have been

identified as carriers of pollutants because of their significant ability to absorb pollutants.[7–9] Hence, they can serve as a representative measure of contaminants (CECs) entering the environment through human activities around informal settlements and ultimately reaching the ocean.[10,11] Occurrence studies supported by new analytical methods are important to inform policymakers, especially for the environmental protection of rivers by preventing the building of informal structures along them.[12–14] The pollution of rivers and subsequently oceans by informal settlements can be monitored during floods by indicators such as microplastics.

Some microplastics are intentionally produced for specific applications, such as in medicine for pharmaceuticals, cosmetic products, equipment used in estuary–sea separation (nets), and cleaning products.[15–18] Microplastics present in the environment cause toxic effects if ingested at high concentrations, such as the disruption of the internal organs and the digestive system of aquatic organisms.[6,19–22] They can cause

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intense effects, such as the potential for cancer, disturbance of the nervous and immune systems, and alterations in reproduction and development.[23–25] As a result, it is crucial to protect the environment from microplastics, which are made up of various polymers such as polyvinyl chloride (PVC), polyester (PE), polypropylene (PPE), polyethylene (PET), polystyrene (PS), polyamide (nylon 6), and acrylic.[26] It is imperative to monitor the occurrence of these microplastics in the environment to understand how to address the issue and contribute additional data to the existing knowledge.[3,14,27]

However, analytical methods for the quantitative extraction of microplastics from sediments are lacking.[28,29] Techniques for sampling the top layer of sediments are crucial, as they result from deposition during flooding. It is imperative to isolate these top layers to obtain distribution data during such events. Several techniques have been proposed for the recovery of microplastics from sediments.[29,30] The most promising analytical method is a density separation technique that employs a saline solution for size separation, followed by sieving and filtration.[31,32] Moreover, the development of new techniques requires microplastic standards for the optimization and validation of these analytical methods. However, matching microplastic standards for specific study areas is either lacking or nonexistent.[33] Consequently, many researchers have prepared microplastic standards from plastic waste, embracing the principles of the circular economy. B. Quinn *et al.* 2017 (ref. 32) employed a single-stage method that utilizes various saline solutions to reliably acquire microplastic polymers of 1 mm or less from sediment samples. The high density of a density separation solution results in an increase in the rate of microplastic recovery. Hence, it was reported that the density of the brine solution should be considered for the high recovery rate of microplastics. Also, A. Käppler *et al.* 2016 (ref. 34) employed the sieving method to separate the particles into different sieves sized between less than 20 μm and above 500 μm. Finally, a size category separation method was implemented in the lower microplastic fraction (500 μm). FTIR imaging was used to analyze particles with a diameter of 500–50 μm in a comparable amount of time and accuracy. Raman imaging was employed for a more detailed and time-consuming analysis of particles sized 50–1 μm. This method proved effective, especially as the amount of microplastics in both salt and freshwater samples increases with decreasing sample size.

This study was undertaken to evaluate the occurrence and distribution of microplastics after floods in Durban, South Africa. Additionally, the role of informal settlements in contributing to transporting loads of contaminants to the ocean during floods was investigated by comparing microplastic distribution in the Mgeni River and Palmiet River. The analytical method developed for this evaluation was based on a density separation method and validated using microplastic standards produced from waste plastic collected upstream of the Palmiet River.

2 Experimental

2.1 Chemicals and reagents

All polymer standards, polyamide (PA), polyethylene (PET), polystyrene (PS), polyvinyl chloride (PVC), polyvinylpyrrolidone

(PVP), polyethersulfone (PES), and polyvinyl sulfate (PVS), were purchased from Sigma Aldrich (Steinheim, Germany). Sodium chloride (NaCl) was purchased from Sigma Aldrich (Steinheim, Germany). All plastic waste was collected from a dumping site in Durban, South Africa. Millipore water was obtained from a Gradient A10 Milli-Q (18 Ω m) system bought from Microsep, South Africa.

2.2 Characterization of microplastics

2.2.1 Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR). FTIR-ATR spectroscopy was carried out on a PerkinElmer Precisely 100 FTIR spectrometer (PerkinElmer, Waltham, MA, USA). The scans were done using a universal ATR with a diamond crystal accessory. The IR spectra for each particle were recorded across a wavenumber range of 400–4000 cm^{-1} with a spectral resolution of 4 cm^{-1}. The background scan, consisting of 90 scans, was recorded before sample analysis. The microplastic material was then placed on the crystal, and force was applied to ensure appropriate contact between the material and the crystal. Spectrum 100 software was used to analyze the spectra.

2.2.2 Stereomicroscopy. A Nikon AZ100 stereomicroscope equipped with a Nikon Fiber Illuminator and NIS-Elements D image software was used. Images were captured using a Nikon DXM1200C camera. Micrograms obtained from stereomicroscopy were used to measure the size of microplastics prepared in this study.

2.3 Extraction method

2.3.1 Microplastic standard preparation. Microplastic fragments were prepared using plastic waste from a dumpsite in Durban, South Africa. The plastic scraps were virgin plastics of different kinds, such as food containers, personal care products, and consumer products. Milling was done using a ball miller operated at a high-energy planetary mill (Retsch PM 400), and the temperature was not controlled under compressed air. Steel 250 mL jars and 10 mm balls were used for milling. A material (10 g) of each plastic waste was used to prepare the fragments under 300 rpm for two hours. The polymeric material present in each product waste was measured using ATR-FTIR before usage in method development.

2.3.2 Density separation. To optimize the microplastic separation method in sediments, two parameters were studied: sediment mass and the volume of the separating solution (NaCl). Sediment masses of 5, 10, 20, and 50 g were mixed with NaCl volumes of 30, 50, and 250 mL. In each experiment, seven pieces of each microplastic fragment were spiked into varying sediment masses and a 1 M NaCl solution. All experiments were conducted in triplicate. NaCl was used to increase the ionic strength; the salt compressed the electrostatic double layer at the microplastic surface, thereby improving the extraction of microplastics from sediments.

The spiked sediments were shaken at 2000 rpm for 3 hours and allowed to settle overnight. Subsequently, they were filtered under vacuum using Whatman filter paper and washed with Millipore water. The recovered plastic fragments in the filter

paper were transferred to a Petri dish and allowed to dry before analysis. Stereomicroscopy equipped with a camera was employed to determine the number and sizes of microplastics recovered. ATR-FTIR was used for spectral analysis.

2.3.3 Sampling. A major informal settlement surrounds the Palmiet River, whereas the Mgeni River passes through mountainous areas away from informal settlements. Informal settlements have been identified as a significant source of microplastic pollution in the ocean during flooding events. Grab sampling was employed to collect samples from five points leading to the sea. Steel shovels were used for collection, and the samples were placed in foil and then dried under a fume hood to avoid contamination. The samples were collected three times at each point, and no plastic equipment was used at any point during sampling and sample preparation. The sampling points are shown in Fig. 1, including the uMgeni River, Palmiet Stream (informal settlement), the joining point of uMgeni/Palmiet Stream, the uMgeni Estuary, and the Sea Point.

2.3.4 Quality control. To prevent the spread of contaminants during the preparation of samples for microplastic analysis, rigorous safety measures were adhered to. The lab environment underwent thorough cleaning and disinfection before commencing sample preparation. All tools and equipment, such as glassware, tweezers, and filtration systems, were either sterilized or washed with suitable cleaning agents to eliminate any potential impurities. All sample pre-treatment was conducted in a fume hood to prevent airborne pollutants. After each run, the milling jars were cleaned using glass followed by acetone to remove any left product to avoid cross contamination. Furthermore, strict quality control protocols were enforced, including the inclusion of blank samples and controls with each batch of samples to monitor and mitigate any potential laboratory-based contamination. Clean Whatman filter papers and NaCl solution were used for quality control.

3 Results and discussion

3.1 Method development

The construction of an in-house database of microplastics was established using plastic waste collected from a dumpsite upstream of the Palmiet River.[28] Different product containers were collected from the dumpsite, including containers for food products, cosmetic products, trash bags, takeaways, and food containers. The source of this garbage can be traced to people living upstream of the informal settlement and the demographic of these people is the same as that downstream. The database was built using the prepared microplastic standards with stereo microscopy and FTIR-ATR. The standards purchased from Sigma Aldrich helped in identifying polymers in the prepared microplastic from waste. These plastic wastes were prepared to align with the properties of microplastics. During flooding, there is the potential for this waste to be washed into the river.[35–37]

3.1.1 Microscopy. The plastic waste was ground with a ball mill to produce smaller sizes equivalent to microplastic ranges. Some fragments were found to be above (mesoplastics/macroplastics) and below (nanoplastics) the size of microplastics and were not used in extraction method development. Confirmation of sizes was performed with a stereomicroscope, and micrograph images with histograms for the length of the

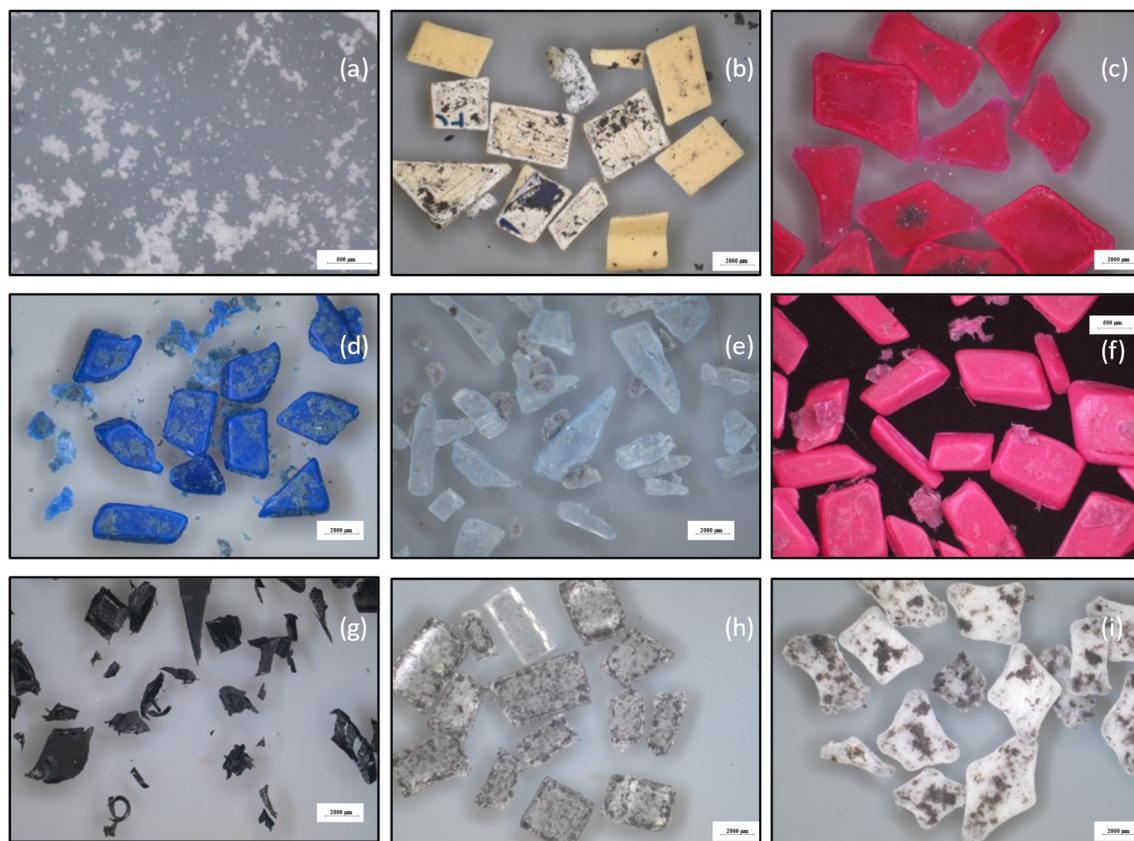**Fig. 1** Sampling map for the studied area, Durban, South Africa.

Fig. 2 Micrographs for the prepared microplastics using a ball miller: plastic takeaway cup (a), margarine (stock) container (b), ice cream cup (c), body lotion Nivea (d), micropipette tip (e), plastic bowl (f), plastic spoon (g), ice tea plastic bottle (h) and peanut butter container (i).

microplastics are shown in Fig. 2. The sizes were further measured using ImageJ software and found to range between 0.74 and 5 mm as a result of sieving through a 5 mm mesh.

3.1.2 Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR). An in-house database was established by examining polymer standards purchased with FTIR. This correlates with the infrared bands identified in the polymer present in the prepared microplastic standards from plastic waste. The polypropylene standard has well-known infrared bands; hence, it was not analyzed but its reference spectrum was obtained from a study done by Chércoles Asensio et al.[38] The positions of the infrared bands differ for each functional group, and this was used to match distinguish polymers present in each product with microplastics.

Furthermore, the analysis of the polymer spectrum for known aliphatic, unsaturated, aromatic, oxygen, carbonyl, and nitrogen peak groups gives the in-house database validity needed for its application in real environmental samples. The polymeric standard results of the absorption bands and the polymers present are presented in Table 1.

The ATR-FTIR spectra for the analyzed polymer standards are shown in Fig. 3, with their respective absorption bands.

The prepared microplastics from plastic waste were correlated with the polymer standards using the IR bands and the present polymers were confirmed as shown in Table 2.

The microplastics were prepared from commonly used plastic products daily in the informal settlement. The polymer standards were employed to confirm the polymer structures

Table 1 Polymer standards analyzed by ATR-FTIR

Polymer standard	Absorption bands (cm^{-1})
Polyamide (PA)	ν –NH– (3298), ν –CH– (2930), O –NC=O– (1635)
Polystyrene (PS)	ν –CH$_2$– (2919), aromatic ring$_{mono}$ (750)
Polyethylene (PET)	ν –CH$_2$– (2963)
Polyvinyl sulfate (PVS)	ν –CH$_2$– (2912), –O(O=S=O)O– (1327)
Polyvinyl chloride (PVC)	ν –CH$_2$– (2912), –CHCl – (607)
Polyvinylpyrrolidone (PVP)	ν –CH$_2$– (3438), –NC=O– (1650), –CN– (1284)
Polyethersulfone (PES)	ν –CH$_2$– (3069), –(O=S=O)R$_2$– (1405), –CO$_{aromatic\ ring}$– (1319), –(O=S=O)$_{aromatic\ ring}$ – (1071)
Polypropylene (PPE)[38]	ν –CH$_3$ (2950 and 2867), –CH$_2$– (2917 and 2838), C–H (–CH$_3$) (1455 and 1375), C–C (1166 and 972)

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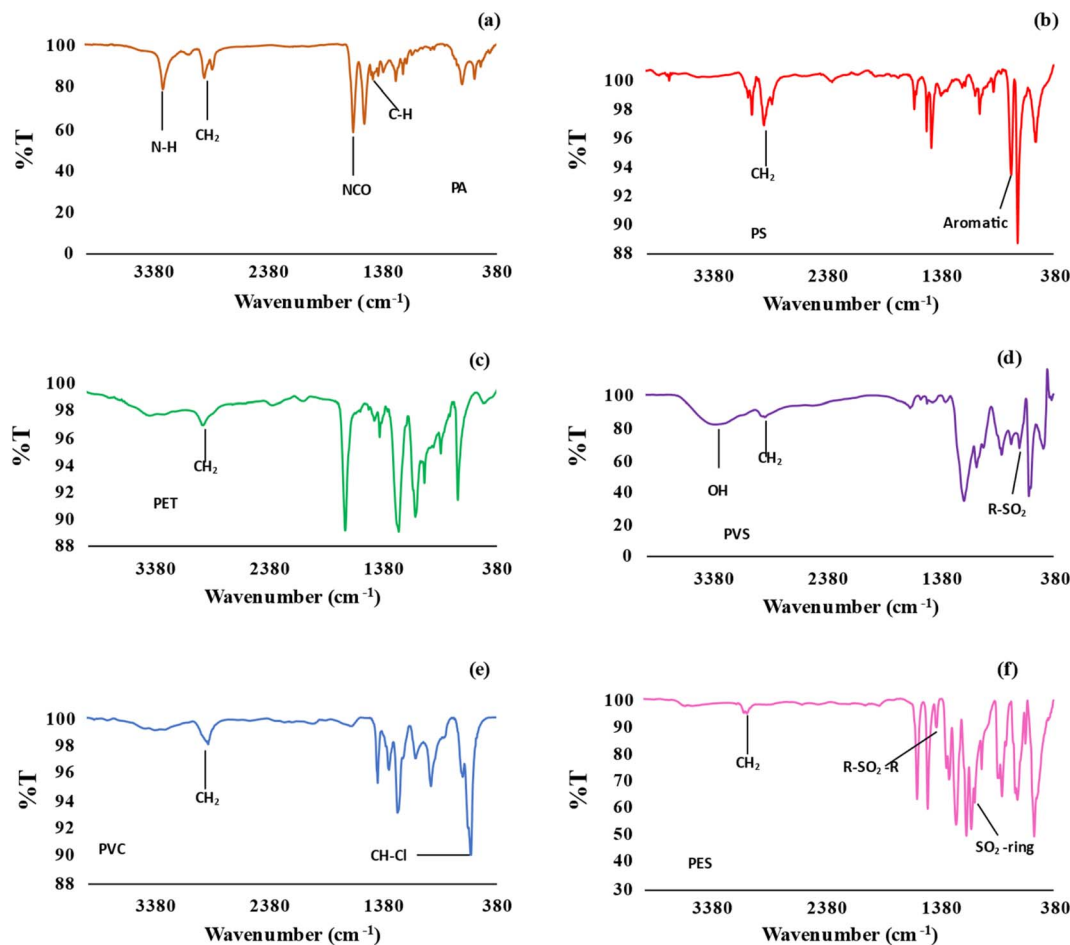Environ. Sci.: Adv., 2024, **3**, 850–860 | **853**

Fig. 3 IR absorption spectrum for PA (a), PS (b), PET (c), PVS (d), PVC (e), and PES (f).

present in microplastics. The margarine (stock) spectrum (Fig. 4a) shows bands: $-CH_3$ (2949 and 2867 cm^{-1}), $-CH_2-$ (2916 and 2838 cm^{-1}), C–H ($-CH_3$) (1458 and 1376 cm^{-1}), and C–C (1167 and 973 cm^{-1}). These features show the properties of PPE. The ice cream cup spectrum (Fig. 4b) was similar to that of PPE as it consisted of the band: $-CH_3$ (2951 and 2875 cm^{-1}), $-CH_2-$ (2917 and 2838 cm^{-1}), C–H ($-CH_3$) (1455 and 1376 cm^{-1}) and C–

C (1166 and 973 cm^{-1}). The ice tea bottle spectrum (Fig. 4c) had a band: aliphatic group, $-CH_2-$ (2963 cm^{-1}), which indicated the presence of PET polymer. A PS polymer spectrum was similar to a plastic spoon spectrum (Fig. 4d) as it shows a band: monosubstituted aromatic ring (750 cm^{-1}), $-CH_2-$ (2917 cm^{-1}), and an alkene (3025 cm^{-1}). The peanut butter container spectrum (Fig. 4e) possesses bands: $-CH_3$ (2950 and 2868 cm^{-1}), $-$

Table 2 Microplastic standards analyzed by ATR-FTIR

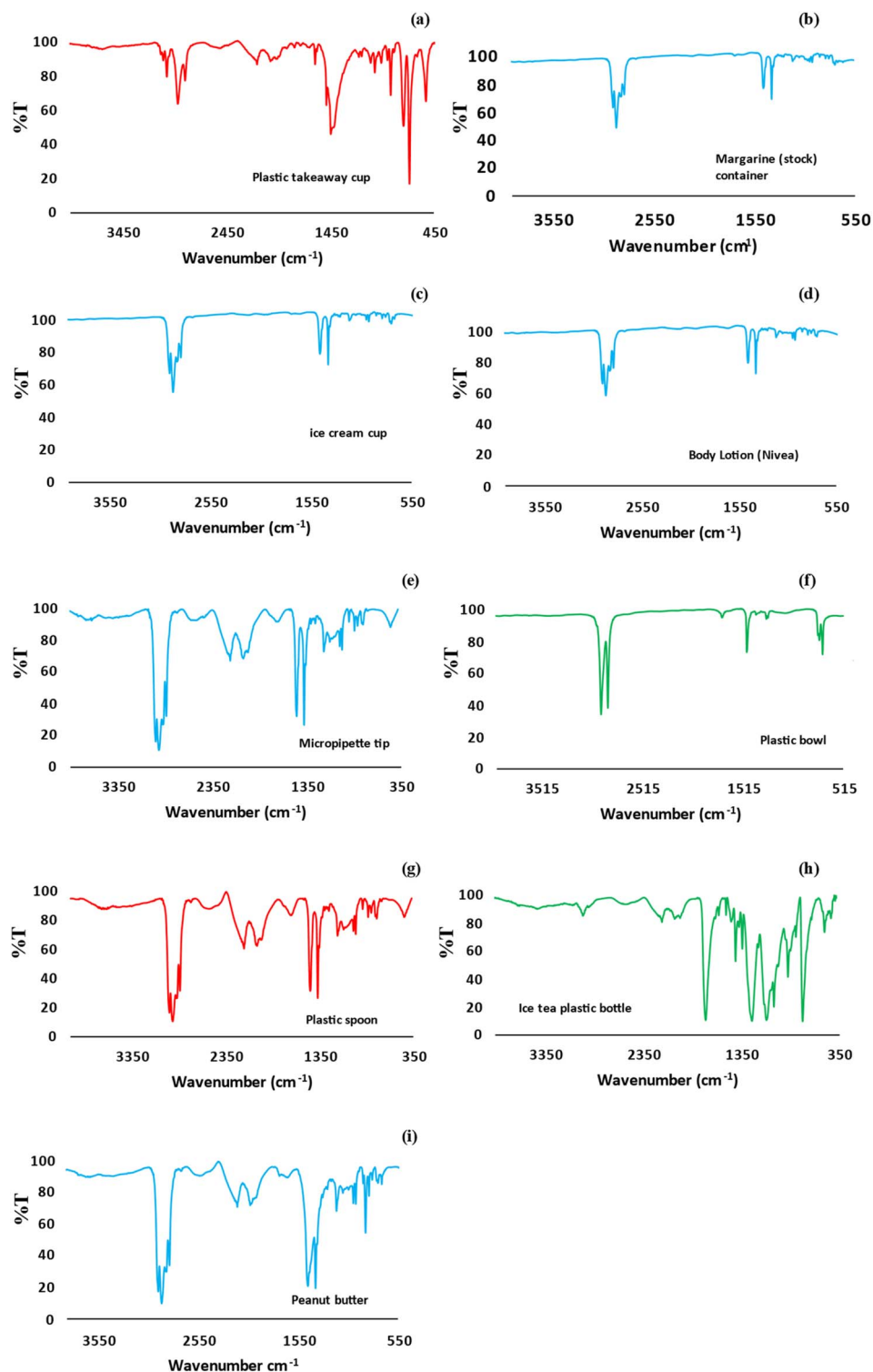|	Microplastic standard	
Plastic waste name	Polymer	Absorption bands (cm^{-1})
Plastic takeaway cup	Polystyrene	ν $-CH_2-$ (2920), aromatic ring$_{mono}$ (752)
Margarine (stock) container	Polypropylene	ν $-CH_3-$ (2949), ν $-CH_2-$ (2916), ν $-CH-$ (2867)
Ice cream cup	Polypropylene	ν $-CH_3-$ (2949), ν $-CH_2-$ (2916), ν $-CH-$ (2867)
Body lotion (Nivea)	Polypropylene	ν $-CH_3-$ (2949), ν $-CH_2-$ (2916), ν $-CH-$ (2867)
Micropipette tip	Polypropylene	ν $-CH_3-$ (2949), ν $-CH_2-$ (2916), ν $-CH-$ (2867)
Plastic bowl	Low-density polyethylene	ν $-CH_2-$ (2915) and $-CH$ (2848)
Plastic spoon	Polystyrene	ν monosubstituted aromatic ring (750), $-CH_2-$ (2917), alkene (3025)
Ice tea plastic bottle	Polyethylene	ν $-CH_2-$ (2963)
Peanut butter container	Polypropylene	ν $-CH_3$ (2950 and 2868), $-CH_2-$ (2916 and 2838), C–H ($-CH_3$) (1451 and 1375), C–C (1166 and 972)

Fig. 4 IR absorption spectrum for a plastic takeaway cup (a), margarine (stock) container (b), ice cream cup (c), body lotion Nivea (d), micro-pipette tip (e), plastic bowl (f), plastic spoon (g), ice tea plastic bottle (h) and peanut butter container (i).

CH_2- (2916 and 2838 cm^{-1}), C–H (–CH_3) (1451 and 1375 cm^{-1}), and C–C (1166 and 972 cm^{-1}), and this shows the functional groups of PPE. The plastic takeaway cup (Fig. 4f) and PS had similar spectra and the microplastic band confirms this: monosubstituted aromatic ring (752 cm^{-1}), –CH_2- (2920 cm^{-1}), and an alkene (3025 cm^{-1}). The micropipette tip spectrum (Fig. 4g)

consisted of PPE functional group bands such as: –CH_3 (2951 and 2875 cm^{-1}), –CH_2- (2917 and 2838 cm^{-1}), C–H (–CH_3) (1455 and 1376 cm^{-1}) and C–C (1166 and 973 cm^{-1}). The plastic bowl spectrum was correlated with the low-density PET as it consists of bands: –CH_2- (2915 cm^{-1}) and C–H (2848 cm^{-1}). Lastly, the body lotion (Nivea) spectrum (Fig. 4h) had a band: –CH_3 (2949

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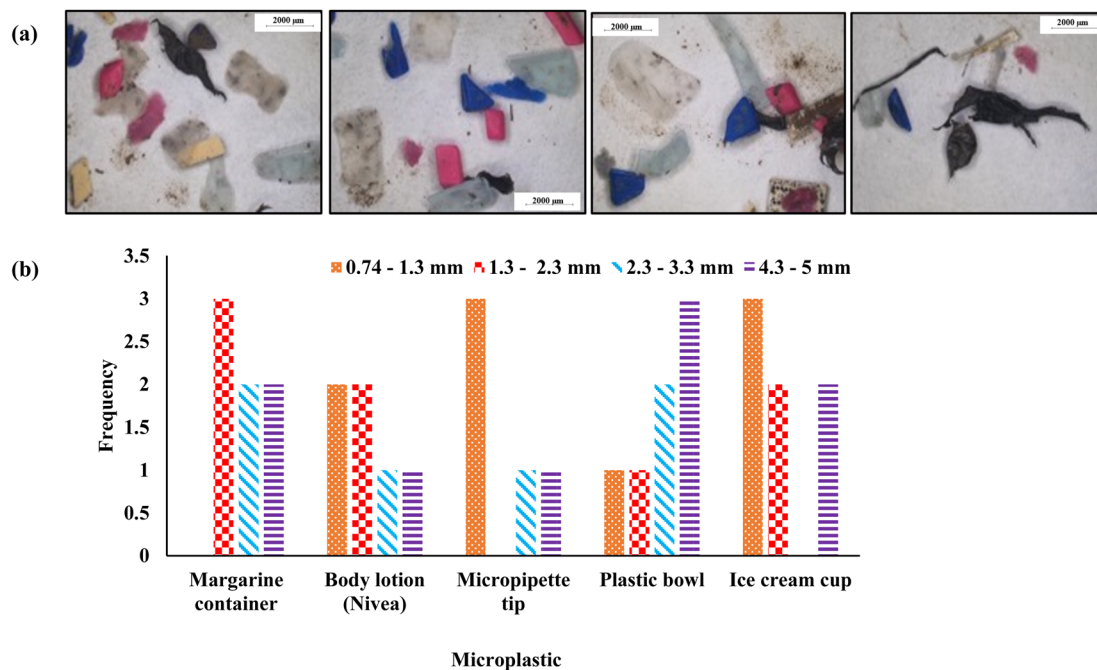Environ. Sci.: Adv., 2024, **3**, 850–860 | **855**

Fig. 5 Stereomicroscope micrographs for the spiked microplastics (a) with the histogram of the recoveries of microplastics (b).
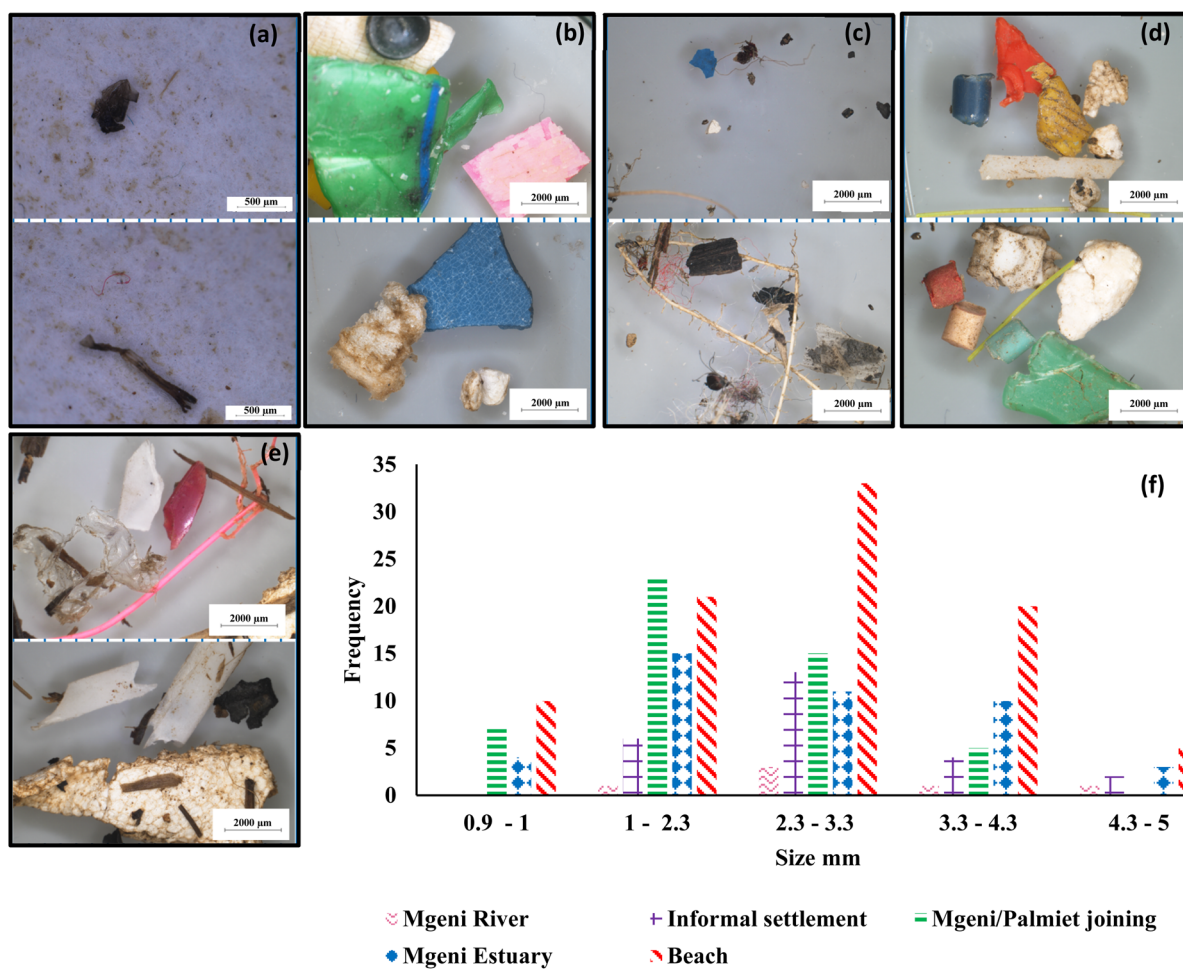

Fig. 6 Micrographs for environmental samples from the Mgeni River before joining (a), informal settlement (Palmiet River) (b), joining Palmiet/Mgeni River (c), uMgeni Estuary (d) and ocean (e) with the histogram for microplastic size (f).

and 2875 cm^{-1}), –CH$_2$– (2916 and 2837 cm^{-1}), C–H (–CH$_3$) (1456 and 1376 cm^{-1}) and C–C (1166 and 973 cm^{-1}), which is similar to PPE.

3.1.3 Density separation method. A density separation method was optimized for the separation of microplastics from sediments. Various amounts of NaCl solution and sediments were used to find the optimum parameters for the separation method. The same amount of 7 strips of the prepared microplastics was spiked in sediments in triplicate, which consisted of different sizes. Then different conditions described in Section 2.3.2 were used for the optimization of the density separation method. A 250 mL NaCl solution with 50 g sediments was chosen as the optimum conditions for separating

microplastics. This was a result of the highest recoveries compared to other conditions used. The recoveries for all microplastics were within 60–120%, as shown in Fig. 5, which is an acceptable analytical range for a good separation/extraction method.[31,32]

3.2 Application to environmental samples

Plastic pollution has increased drastically in the marine environment due to recent floods in KwaZulu-Natal, South Africa. This research demonstrates that informal settlements are a contributor of microplastics to the ocean. Stereomicroscopy and ATR-FTIR were used to confirm the occurrence of different microplastics in sediments. In the informal settlement, three
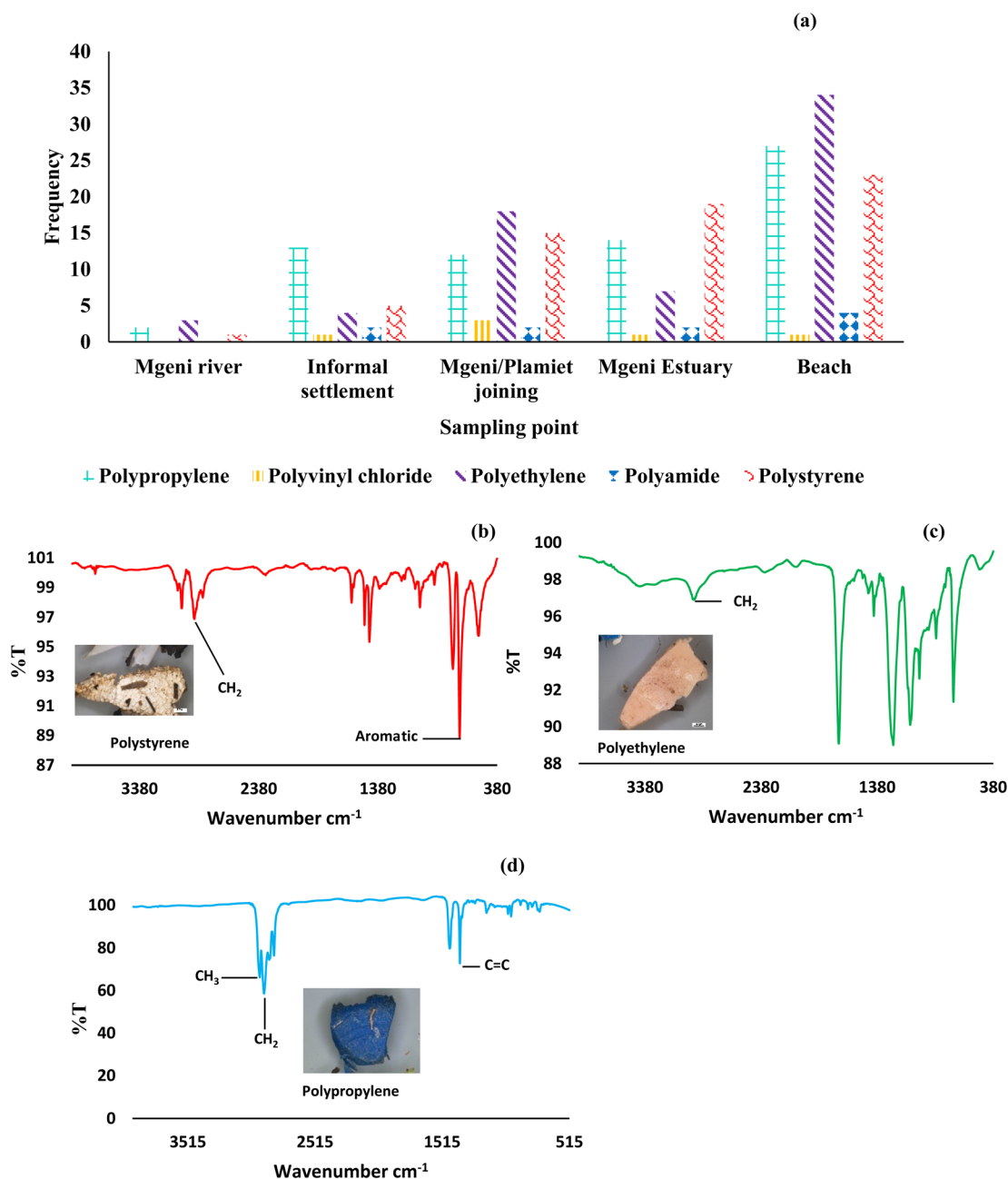

Fig. 7 Histogram for environmental samples from the Mgeni River (a) and spectra for (b) polystyrene, (c) polyethylene, and (d) polypropylene.

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Table 3   Comparison of microplastic occurrence from the literature with the current study

Sample type	Location	MP abundance	Shape	Dominant colour	MP type	Reference
Sediments	Western Cape, South Africa	185	Fragment, filament, sphere, film, foam	White, black/grey, blue/green, yellow, transparent	PET, PE, PS, PAA, PP, SSR	D. Julius et al.[41] (2023)
Sediments	South China	103 080	Fiber	Transparent	PE, PP, PET	Y. Wan et al.[42] (2022)
Surface water	Kenya	1473	Films, fiber, fragments	White, blue and green, black, red, transparent, blue, multicolor	PP, HDPE, LDPE	Kosore et al.[43] (2022)
Sediments	Eastern Cape, South Africa	213	Fiber, fragment	Yellow, pink-red, black, transparent, blue, green, black	PP, PE, PS	Current

samples were taken and were made into a composite sample, and this sampling area shows the presence of different sizes of microplastics with a size of 0.96–5 mm (Fig. 6b), with the uMgeni River showing the least amount of microplastics with size between 1 and 5 mm (Fig. 6a). This shows that most of the plastics were not found in the uMgeni River before informal settlement, but other streams passing through the informal settlement might be the source. The joining point had microplastic size from 0.91 to 5 mm (Fig. 6c); the presence of microplastics less than 1 mm shows years of pollution as microplastics slowly break in the environmental matrices rather by the current of the fast-moving river during the floods.[39,40] The Umgeni Estuary had a high volume of microplastics with size ranging from 0.98 to 5 mm (Fig. 6d); this is due to microplastics being transported from the informal settlement to the sea. The microplastics were washed down during the flood that occurred recently in the studied area, and most of the microplastics were found at the beach with a size of 0.90–5 mm (Fig. 6e); this point was found to be the most polluted by microplastics. Most of the microplastic fragments found at the sea were similar in polymer type to those in the informal settlement.

The microplastics found on different points were analyzed using ATR-FTIR. Polymeric types for example PET, PS, PA, PVC, and PPE were found on microplastics in all sampling points except for the uMgeni River. At the uMgeni River, only three polymers were found, polypropylene, polystyrene, and polyethylene. These three polymers were the most abundant in microplastics obtained in the studied area (Fig. 7a); this is a result of many plastic products used in households being made of similar polymers.

This is an indication that many plastic products used in households are made of these similar polymers. The IR spectra and the micrographs of the most abundant polymers are shown in Fig. 7b (PS), 7c (PET), and 7d (PPE). All microplastic polymers found at the informal settlement point were also present at the sea; this may be due to the washdown during the floods.

3.3   Comparison of the current study with the literature

A comparison of MPs found in informal settlements in this present study with some reported in previous studies is presented in Table 3. In comparison to previous studies conducted in informal settlements, the microplastics identified exhibited a variation in characteristics. These microplastics were observed with a diverse array of colors, types, and shapes, indicating a broader range of sources and potential pathways for contamination. Furthermore, the abundance of microplastics in both sediments and water samples differed significantly, suggesting a potentially more complex and pervasive presence of these pollutants in the environment of informal settlements. This variability underscores the need for comprehensive and targeted strategies to address the multifaceted challenges posed by microplastic pollution in such areas.

4   Conclusion

In the present study, microplastic standards matching the study area were successfully prepared using ball milling. These standards contained particles ranging between 0.74 and 5 mm, as confirmed by stereomicroscopy. The method employed did not alter the chemical or structural properties of polymers present in plastic waste. The identified polymers in various products included polystyrene, polypropylene, polyethylene, and low-density polyethylene, all confirmed by FTIR. The prepared microplastic standards were then utilized to optimize a density separation method for sediments. The percentage recoveries were consistently above 60% for the selected parameters, specifically when using 250 mL of 1 M sodium chloride as the separation solution in 50 g of sediments. This optimized method was further employed to investigate the occurrence of microplastics in environmental samples. The recent floods revealed a significant impact on marine plastic pollution originating from informal settlements. This was evident through the recovery of microplastics from the sea, attributed to the informal settlement. The size range of the detected microplastics on the environmental samples was 0.90–5 mm. Furthermore, the ATR-FTIR analysis confirmed the presence of the most common polymers in the studied area, namely polystyrene, polyethylene, and polypropylene.

Author contributions

Nokwanda Hendricks: conceptualization, methodology, formal analysis, writing – original draft, and investigation. Olatunde S.

Conflicts of interest

The authors declare herewith that they do not have any conflict of interest with any organization or individual.

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