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Microplastic–metal nexus in soil and freshwater fish: distribution, bioavailability and risk assessment

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ABSTRACT

This paper examined the prevalence of microplastics (MPs) and toxic metals (TMs) in soils and freshwater fish in three urban centres, Osogbo, Ilesa and Ile-Ife, in southwestern Nigeria. Soil and fish were collected during the wet and dry seasons, extracted and characterised using density separation, oxidative digestion, Fourier Transform Infrared Spectroscopy (FTIR) and optical microscopy. An inductively coupled plasma optical emission spectrometer (ICP-OES) was used to quantify metal content. Contamination models, estimated daily intake (EDI), hazard quotient (HQ) and lifetime cancer risk (LCR) were used to measure ecological and human health risks. The MP abundance in soil ranged from 1,200 to 5,733 particles/kg, and in fish from 1,045 to 130,556 particles/kg, with the wet season showing the highest values. The trend of MPs in fish organs from different rivers follows: GIT > liver > muscle > kidney > heart. Elevated levels of Fe, Al, Zn, and Cu were found in the metal analysis, along with toxic metals Pb, Cd, Cr, and Ni, with Cd posing the greatest ecological concern. The result also showed significant non-carcinogenic and carcinogenic risks associated with toxic metals, particularly Cr, Pb and Cd. The EDI of MPs in fish organs showed a significant seasonal variation, with exposure substantially higher during the dry season.

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Introduction

In developing countries, contamination from plastics and toxic metals (TMs) is increasingly becoming a major environmental concern, driven by high population density, rapid urbanisation, industrial expansion and inadequate waste management practices. These factors collectively pose significant pressure on both human health and environmental sustainability [1]. It is estimated that approximately 33% of global plastic production is disposed of in landfills or freshwater systems under poorly managed conditions [2]. In such environments, plastics undergo progressive degradation, releasing hazardous substances into ecosystems [3]. These materials break down due to ultraviolet (UV) radiation, hydrolysis, mechanical abrasion and microbial interactions [4, 5], resulting in microplastics (MPs), which are usually 5 mm to 1 mm. Further degradation results in the formation of nanoplastics (NPs), which are less than 1 µm [6].

Soil is one of the most important abiotic factors in terrestrial ecosystems, which helps maintain plant growth and biodiversity. It is an important resource of agricultural productivity and ecological balance [7]. Nonetheless, soil pollution from MPs originating in plastic mulching, pharmaceutical residues, cosmetic products, tyre wear particles, textile fibres, and the improper disposal of plastics has become a major

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environmental concern [8, 9]. When incorporated into the soil matrix, MPs may impact negatively on soil functionality and plant development through the change in physicochemical properties resulting to slowed seed germination, decreased soil bulk density, impaired root penetration, diminished aeration and release of toxic plastic-derived leachates, all of which have adverse effects on soil microbial communities needed for plant health [10, 11, 12, 13].

Additionally, the presence of MPs can disrupt essential soil characteristics, including moisture retention, water-holding capacity, pH, and porosity, which ultimately affect organisms that rely on them [14]. Beyond the structural alterations, MPs may have toxic impacts on soil biota, such as plants, invertebrates and microorganisms, and thus interfere with the operation of the ecosystem [15, 16]. Although the uptake of MPs by plant cells is usually low due to their large size and surface properties, NPs can enter root cell walls and alter cellular functions, thereby influencing plant growth and development [17, 18]. Likewise, MPs readily adsorb TMs owing to their negative surface charges and a wide range of functional groups [19]. As metal carriers, MPs may contribute to the distribution of accumulated TMs along the food chain, raising the possibility of human exposure [20]. Van der Waals forces, complexation, electrostatic attraction, and physical adsorption all contribute to the complex process of TM adsorption onto MPs [21].

Notably, MPs alter the sorption/desorption processes in TM-contaminated soil, increasing the accessibility and bioavailability of metal ions. Once TMs accumulate in plant tissues, they disrupt critical physiological and biochemical processes, ultimately affecting plant development and production [22, 23]. The coexistence of MPs and As(III) has been linked to elevated levels of peroxides and reactive oxygen species (ROS), which may disrupt the cell membrane [24]. Likewise, [25] established that concomitant exposure to MPs and cadmium (Cd) had a significant negative effect on shoot and root length and stem diameter in strawberry plants. [26] also revealed that polystyrene (PS), when combined with copper (Cu) and lead (Pb), exhibits synergistic toxicity in rapeseed plants, as evidenced by elevated malondialdehyde levels, indicating oxidative stress. Moreover, exposure to MPs and TMs has been associated with various negative health effects, such as growth retardation, cancer development, kidney disease, respiratory diseases, gastrointestinal blockage and death [27].

More worrisome is the ingestion of MPs by aquatic organisms, which often mistake them for food [28]. This results in exposure to a wide variety of hazardous contaminants, including persistent organic pollutants (POPs) such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), dichlorodiphenyltrichloroethane (DDT), and TMs that adsorb onto plastic surfaces [29]. In Nigeria, fish contribute more than 60% of total protein consumption; therefore, they are a major route of exposure to MPs and their associated pollutants [30]. Upon ingestion, these contaminants are bioavailable and can cause adverse biological effects, including metabolic and reproductive dysfunction, immunosuppression, oxidative stress, cellular and subcellular damage, inflammation, and carcinogenesis [31, 32]. Humans are not an exception, as fish are among the largest sources of dietary protein worldwide [33, 34, 35].

Given these associated human and environmental risks, it is imperative to comprehensively examine the combined effects of MPs and TMs across terrestrial and aquatic ecosystems to mitigate their impacts and safeguard human and environmental health.

Study area

Three main urban centres in Osun State, Nigeria—Osogbo, Ilesa, and Ile-Ife—were used to collect soil samples, while the three rivers nearest the sites where the soil samples were collected were used to collect fish samples (Figure 1).

The state capital, Osogbo, has an average population of 822,497 [36]. It is bounded by Ede, Iragbiji, Ilesa, and Ikirun. Osun-Osogbo is not only the administrative hub but also the location of the yearly Osun-Osogbo festival and a UNESCO World Heritage site [37]. The Osun River is one of the major rivers in southwestern Nigeria, flowing through five states and finally emptying into the Lekki Lagoon, which leads to the Gulf of Guinea (Atlantic Ocean) via the Lagos Lagoon. The river sustains a variety of aquatic life, especially tilapia, which are trapped and sold by local fishermen [37]. Nonetheless, the Osun River is susceptible to both geogenic and anthropogenic contaminants [38]. The three soil sampling locations in Osogbo were, Stadium Roundabout (7.78002° N, 4.54847° E), Ofa Bolanle Roundabout (7.78956° N, 4.50650° E) and Old Garage Roundabout (7.79336° N,

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4.57328° E) (Figure 1a), whereas, fish were sampled from Osun River (7.79611° N, 4.62306° E) (Figure 1b).

The population of Ilesa, the second sampling location, is predicted to be 431,992 [37]. Due to its gold mining operations, which have boosted traffic, particularly from trucks carrying the ore, it is economically significant. The soil sampling point was Ilesa Roundabout (7.62142 ° N, 4.74005 ° E) (1a), whereas fish samples were collected from Oora River (7.66028 ° N, 4.8825 ° E) (Figure 1b). Ile-Ife is the third sampling location with a population of 438,074 (WPR, 2024). The city hosts of two universities and a large recycling plant. Soil was sampled at Mayfair Roundabout (7.48908 ° N, 4.53519 ° E) (Figure 1a), whereas fish were collected from Opa River (7.56000 o N, 4.71472 ° E) (Figure 1b).

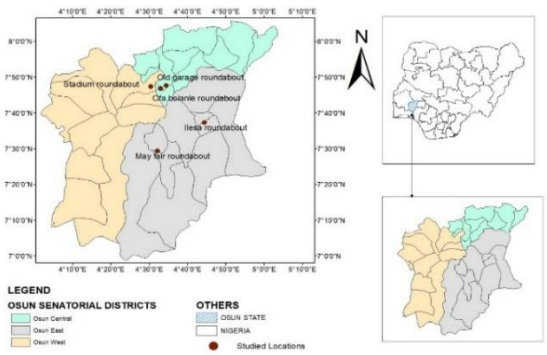

Fig. 1a Map of Osun State, Southwestern Nigeria, showing the soil sampling locations in red

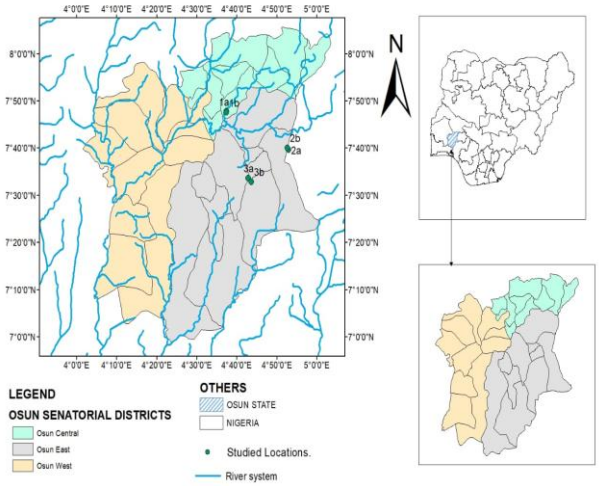

Fig. 1b Map of Osun State, Southwestern Nigeria, showing the fish sampling locations (1-Osun River, 2-Oora River and 3-Opa River) in green

Methodology

Soil sample collection

In five significant intersections in Osogbo, Ilesa, and Ile-Ife, soil samples were taken using a stainless-steel soil auger at a depth of 10 cm in accordance with the protocol previously described by [38]. To obtain a representative sampling, triplicate sub-samples (1 kg) were collected at each sampling location and combined. In order to obtain comprehensive composite sampling, sampling was conducted during wet and dry seasons. After a number of preliminary steps, such as mixing and air-drying, the composite samples were sieved through a 5 mm mesh sieve to remove stones and other unwanted components.

Sample analysis and quantification of MPs in soil

Soil samples (10 g) were examined in triplicate using the procedure outlined by [39]. Laboratory-grade stainless-steel sieves with pore diameters of 53, 75, and 125 µm were used to successively sieve each sample in ascending order. For further processing, the residual fractions from each sieve were transferred into pre-cleaned beakers. Each sample was treated with 40 mL of 30% hydrogen peroxide (H_2O_2) to eliminate organic materials. To ensure complete organic material digestion, the mixtures were shaken for 2 hours at 300 rpm and 27 °C using a UNISCOPE shaker (Model SM101, Surgifriend Medicals, England). They were then left to stand for four days. Following pressure filtration through 0.45-µm pore-size filter paper, the samples were oven-dried at 50 °C. The dried residues were carefully scraped into pre-cleaned conical flasks for density separation.

Density separation was accomplished by mixing the residues with 200 mL of a 5 M sodium chloride (NaCl) solution. The mixtures were covered with aluminium foil, shaken for five minutes, and then left for a whole day. Low-density MP-containing supernatant was decanted, and the residue was vacuum-filtered. The procedure was repeated after treating the residual with 50 mL of 0.1 M zinc chloride ($ZnCl_2$) solution to further isolate MPs. After being recovered from density separations with NaCl and $ZnCl_2$, the MPs were added, oven-dried for 24 hours at 50 °C, and kept in glass Petri dishes for examination. An optical microscope (Model DM-125+MD101, China) with a digital camera was used to study shapes and colours at a magnification of 40×. Polymer recognition was conducted using Fourier Transform Infrared (FTIR) spectroscopy (Shimadzu FTIR-8400S, China) to determine the characteristic functional groups of MPs.

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Fish sample collection

Freshwater fish (*Oreochromis niloticus*) with a total length of 25.0-33.2 cm and a weight of 0.38 to 0.75 kg (wet weight) were sampled in triplicate with the help of local fishermen from Osun, Oora and Opa Rivers. The fish samples were rinsed with tap water, then with deionised water, to remove surface contaminants. The samples were placed in ice-packed coolers and then identified at the Department of Animal and Environmental Biology, Osun State University. Sampling was conducted during the wet and dry seasons. Following the successful identification, the fish samples were excised, and the selected internal organs were weighed.

Analysis and quantification of MPs in fish samples

Following the procedure previously described by [40], fish organs, including the gastrointestinal tract (GIT), liver, kidney, heart and muscle, were subjected to oxidative digestion. Each organ sample (in triplicate) was placed in separate 250 mL pre-cleaned glass beakers and digested with 20 mL of 10% hydrogen peroxide (H_2O_2). The mixtures were heated on a hot plate at 50 °C for 15 minutes, then incubated at 25 °C with agitation at 300 rpm for 20 minutes. Thereafter, an additional 20 mL of 30% H_2O_2 was added to each sample, followed by 20 mL of 0.1 M aqueous iron (II) sulfate ($FeSO_4$) to enhance the oxidative digestion process. The samples were allowed to cool, covered with aluminium foil to prevent cross-contamination and left for five days, followed by pressure filtration using 0.45 µm pore-size filter paper. The residues were oven-dried at 50 °C and stored in pre-cleaned glass Petri dishes. For the identification and quantification of MPs in fish organs, a digital microscope with an attached camera (Keyence VHX2000, Mechelen, Belgium) was used to assess colour and shape, and quantification was performed manually. The total particle abundance was expressed in particles/kg.

Analysis of metals content

Soil and fish organs were digested for metal analysis using the technique previously described by [41]. An inductively coupled plasma-optical emission spectrometer (ICP-OES) was used to measure the target metal concentrations. For Cd, Co, Cr, Cu, Mn, Ni, Pb, Zn, and Fe, the instrument showed detection limits of 0.1, 0.4, 0.8, 0.6, 0.5, 1.0, 0.3, and 1.0 µgL-1, respectively. The majority of the metals of interest had calibration plots with a coefficient of determination (R^2) of 0.99.

Ecological risk assessment

The ecological risk factor and risk index were calculated for ecological risk assessment of TMs associated with the soil and fish samples using Eqs. (1), (2) and (3) as previously reported by [42].

$$Cf = \frac{C_i}{C_0} \quad (1)$$

$$ERf = (Cf_i \times TR) \quad (2)$$

$$RI = \sum Erf \quad (3)$$

Where Cf represents the contamination factor, Ci represents the measured concentration of metals, Co represents the reference concentration of metals, TR is the metal toxic response, Erf is the ecological risk factor, and RI is the potential ecological risk index.

Health risk assessment

Carcinogenic and non-carcinogenic risk were estimated in soil and fish samples using equations (1), (2), (3), (4) and (5) as previously reported by [43]

$$DI = \frac{Cmetal \times IR}{Bw} \quad (1)$$

$$ADD = \frac{DI \times Ef \times DE}{Tp} \quad (2)$$

$$HQ = \frac{ADD}{RfD} \quad (3)$$

$$HI = \sum HQs \quad (4)$$

$$LCR = ADD \times CSF \times LT \quad (5)$$

where Cm represent metal concentration, IR-ingestion rate, DI- Daily intake of metal, Ef-exposure frequency, DE- duration of exposure and time period over which dose is averaged, R_fD-Reference dose, CSF- carcinogenic slope factor and LT- average lifetime.

Quality assurance

The laboratory benches were thoroughly cleaned with 10% ethanol during the extraction phase, and all glassware was washed with distilled water before being rinsed with an HNO_3 solution. To avoid cross-contamination, samples were wrapped in aluminium foil. Blank was transported to the field, digested, and examined for MPs and TMs identical to those found at the sample sites. The serial dilution approach was used to obtain suitable concentrations of working standards from the multi-element stock standards for metal analysis.

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Moreover, all windows and doors were closed. White cotton laboratory coats, single-use latex gloves and facemasks were used throughout the sampling phase to prevent atmospheric deposition of plastic particles. The ICP-OES expert software was used to create a new worksheet in which the method, its parameters, and each sample code were entered. Following the instrument's programming, the standards were run to obtain the calibration curves, and the samples were then examined in accordance with the sequence parameters in the newly constructed worksheet. For Cd, Co, Cr, Cu, Mn, Ni, Pb, Zn, and Fe, the instrument showed detection limits of 0.1, 0.4, 0.8, 0.6, 0.5, 1.0, 0.3, and 1.0 µgL-1, respectively. The majority of the metals of interest had calibration plots with a coefficient of determination (R^2) of 0.99.

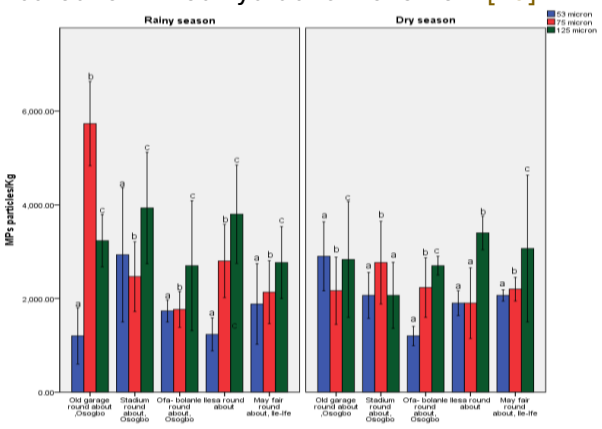
Statistical analysis

The mean ± standard error of the mean (SEM) of three replicates is used to express the study's results. A post hoc Duncan multiple range test was used after a one-way analysis of variance (ANOVA) to compare the means of MPs and TMs in SPSS 2020.

Results and discussion

MPs abundance, shapes and colours

The MP abundance in soil varied seasonally across the sampling sites (Figure 1a). During the dry season, the abundance varied between 1,200 MP particles/kg at the Ofa-Bolanle roundabout and 3,400 MP particles/kg at the Ilesa roundabout. Whereas, the higher concentrations were recorded during the wet season, with an abundance of 1,233 MP particles/kg at the Stadium roundabout and a maximum of 5,733 MP particles/kg at the Old Garage roundabout. The significantly high MP abundance at Old Garage is likely due to high population density, heavy traffic congestion, and the indiscriminate disposal of plastic waste into drainage [44]. The most dominant MP size was 125 µm (39 %), followed by 75 µm (35%), particularly at the old garage roundabout. The predominance of larger particle fractions (125 µm and 75 µm) suggests that urban soils can serve as reservoirs for secondary MPs generated through mechanical fragmentation and photo-oxidative degradation of macroplastics [45]. Conversely, 53 µm MPs were recorded at a lower abundance (26%), suggesting increased leaching of MPs into the soil matrix [46]. Moreover, increased MP abundance in the rainy season is associated with runoff-mediated transport linked to poor drainage systems [47], whereas decreased levels in the dry season may be attributed to limited hydraulic movement [48].



Data expressed as mean ± standard error of mean three replicate concordant values. Data with different superscripts are significantly different at p < 0.05

Fig 1 MPs abundance in soil samples

The MP colours identified in the soil included white, black, blue, red, brown, yellow, green and purple (Figure 2). The most prevalent were white-coloured MPs, especially at Stadium and Old Garage Roundabouts, where they were abundant during the rainy season. Black MPs were the second most dominant colour, and their concentrations were highest at high-traffic locations such as May Fair and Ilesa Roundabouts. The other colour types were found in lower proportions, at less than 15% of the total abundance. The prevalence of white-coloured MPs can be attributed to the intensive use and degradation of single-use plastic packaging materials such as low-density polyethene (LDPE) [44]. Furthermore, the increase in white MPs could also result from photo-oxidative bleaching, in which pigment loss occurs due to extended exposure to sunlight [49]. Conversely, the large number of black MPs is attributed to the wear of vehicle tyres and the deterioration of industrial rubber products [45]. Notably, MP colour affects metal bioavailability and toxicity [50].

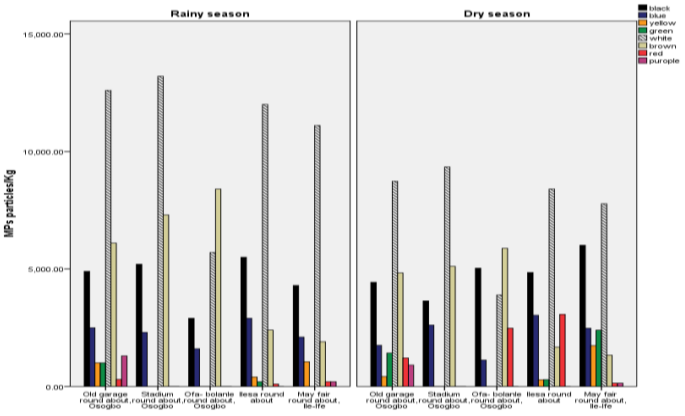


Fig 2 Colours of MPs in the soil sample

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Three different MP shapes, including fibres, fragments and microbeads, were found at all sampling locations (Figure 3). Fibres were the most prevalent in both seasons, followed by fragments, both constituting more than 90 % of the total MP abundance. The highest abundance of the fragments was observed at Old Garage Roundabout and fibres at Stadium Roundabout. Conversely, microbeads were less abundant. They are associated with plastics, which are less used and do not easily degrade in the environment [47]. The high percentage of fibre is attributed to degradation of domestic and commercial textiles due to long-term exposure to ultraviolet [51]. The shape of MPs has a profound effect on biological interactions; fibres can be ingested by soil organisms, whereas fragments can cause intestinal lacerations or tissue damage [52].

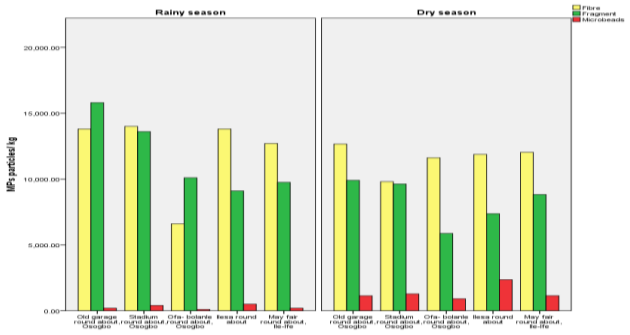


Fig 3 Shapes of MPs in soil samples

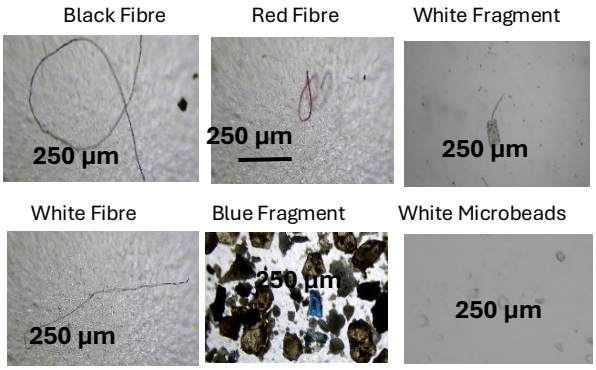
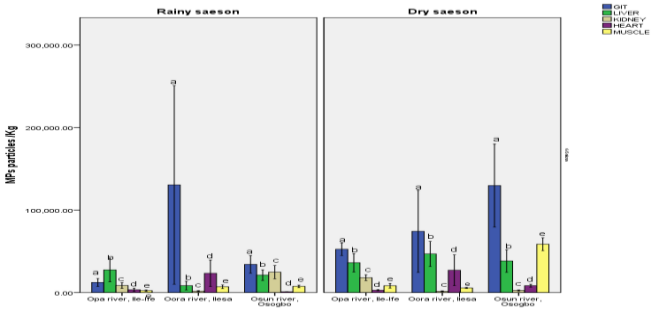


Fig. 4 Selected micrographs of MPs identified in soil and fish organs across different sampling locations

Figure 5 showed the abundance of MPs in fish organs, GIT, liver, kidney, heart and muscle across sampling sites. The abundance was organ-specific, with GIT showing the highest MPs in fish in Osun, Oora and Opa Rivers. MPs were also observed in the liver and kidney, especially in fish from the Osun River, whereas the heart and muscle showed lower abundance. Seasonal change showed that fish in Oora and Osun Rivers had higher MP abundance in the GIT and liver during the rainy season, suggesting greater exposure and uptake. Conversely, fish in the Opa River had lower MP abundance, implying minimal anthropogenic contribution. Ingestion is the primary route of exposure, as evidenced by the presence of NPs in the GIT [53, 54]. The presence of MPs in internal organs suggests that they can be translocated through the intestinal epithelium [55]. Their presence in muscle tissues also raises food safety concerns, since MPs can cause oxidative stress and DNA damage [29, 56].



Data expressed as mean ± standard error of mean three replicate concordant values. Data with different superscripts are significantly different at $p < 0.05$

Fig 5 Abundance of MPs in fish organs

Figure 6a and 6b showed the distribution of MP colours in fish organs. Black-coloured MPs were prevalent in all the sampling sites and organs, particularly during the rainy season. The second most common fraction was white MPs. The other colours, such as blue, pink, yellow, and green, were also observed, indicating a variety of sources for plastic additives and dyes. The GIT had a significant concentration of black, whereas MPs were also detected in muscle tissues, particularly in fish of the Osun River during the dry season. The high concentration of black MPs is linked to tyre and brake wear, while white MPs are possibly from the degraded LDPE [44]

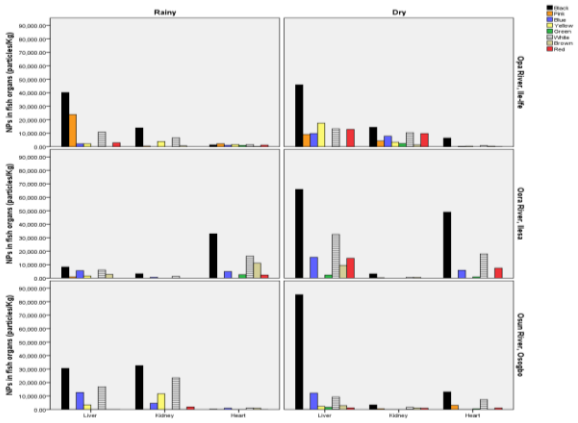


Fig 6a Colours of MPs in fish organs (Liver, kidney and heart)

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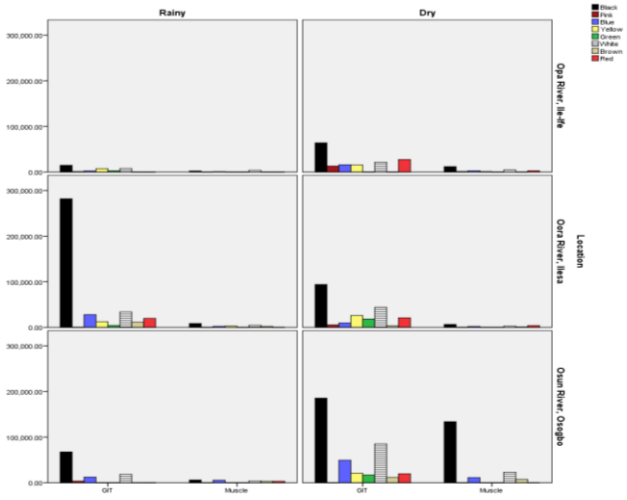

Fig 6b Colours of MPs in fish organs (GIT and muscle)

Figure 7 showed the distribution of MPs shapes across fish organs, with fragments predominating, followed by fibres, while microbeads were least abundant. The GIT exhibited the highest abundance, whereas liver and kidney also accumulated notable amounts. In contrast, the muscle and heart contained fewer particles, mainly small fragments, indicating organ-specific variation influenced by exposure pathways and physiology. The dominance of fragments reflects extensive environmental degradation of larger plastics into secondary particles [57, 58]. Their irregular structure enhances surface area and contaminant adsorption, increasing ecological risks. Fibres, consistently detected across all organs, likely originate from textile and wastewater sources and are easily ingested and retained [59]. Their presence in internal organs suggests translocation from the GIT, as reported in previous studies [60].

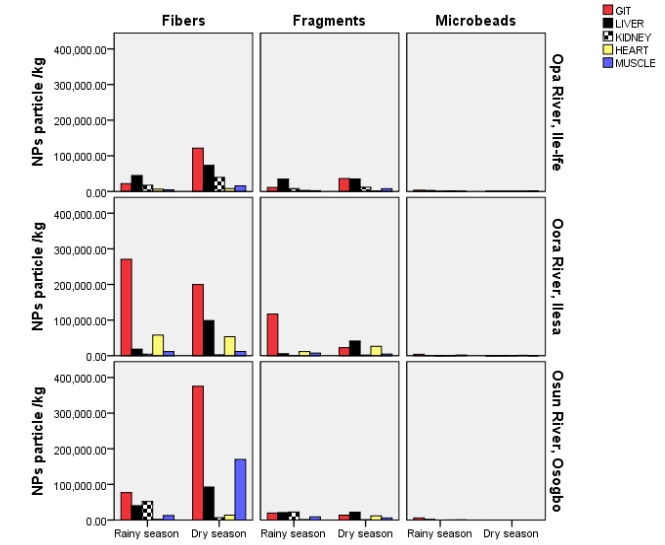

Fig 7 Shapes of MPs in fish organs

Elemental contents of soil and fish samples

The concentrations of metals linked to MPs in the soils of the old garage, stadium, Ofa-bolanle, Ilesa, and May Fair roundabouts during the dry season are compiled in Table 1. Al and Fe predominated in the majority of these sites, with levels ranging from 4517 mg kg^{-1} (May fair) to 13,988 mg kg^{-1} (Ofa-bolanle) and 18,864 mg kg^{-1} (Stadium), respectively. There was also an increase of Ca and Mg, especially in Ofa-bolanle, which implies a high mineral enrichment likely caused by geogenic supplies. Cd was not detected in Mayfair and was highest in Ofa-bolanle (2.120 mg kg^{-1}). Cr was highest in Ilesa (82.731 mg kg^{-1}), Cu in Ofa-bolanle (96 mg kg^{-1}) and Pb in old garage (66 mg kg^{-1}), whereas Zn in Ilesa and Stadium was fairly high. A similar pattern was observed with other metals (Ni, Co, Mn, V, Na, K), with higher concentrations being recorded at Ofa-bolanle and Ilesa roundabout. These high concentrations especially of Cd, Pb, Cr, and Cu is indicative of anthropogenic influence from automobile, waste disposal and urban runoff. The toxicity of metals, particularly Cd and Cr endangers the ecological and human health because it is persistent, bioaccumulated and even carcinogenic [50].

Table 2 summarizes the concentrations of metals associated with MPs in soils from old garage, Stadium, Ofa-bolanle, Ilesa and May fair roundabouts during the rainy season. There was significant reduction in concentration compared to the dry season. Al and Fe were also prevalent, with Al ranging from 930 mg kg^{-1} (Ilesa) to 4710 mg kg^{-1} (Stadium), while Fe peaked in Stadium (16,367 mg kg^{-1}). Ca and Na were also relatively high, particularly in old garage roundabout, suggesting notable mineral inputs. TMs showed distinct site-specific patterns. Cd was highest in stadium (1.260 mg kg^{-1}) and lowest in Ofa-bolanle, while Cr peaked in Stadium but was negligible in Ofa-bolanle and Ilesa. Cu and Pb were highest in old garage, whereas considerably lower in Ofa-bolanle and below detection in Ilesa roundabouts. The decline in metal concentrations, reflects rainfall-driven processes such as leaching, runoff, and dilution, which enhance the mobility and redistribution of MPs and associated metals. Additionally, changing environmental conditions during rainfall may promote desorption of metals from MPs, facilitating their transport. The continued presence of TMs, particularly Cd, underscores ongoing ecological and human health risks due to their mobility, persistence, and bioavailability.

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Table 1: Metal concentrations associated with MPs in different soil samples (Dry season) (mgkg^{-1})

Metals	OGS	STD	OFS	ILSS	MAFS
Al	6936 ± 0.178^{c}	6934 ±0.335^{b}	13988± 0.672^{e}	8255± 0.400^{d}	4517 ± 0.391^{a}
Ba	78.68 ± 0.004^{d}	48.540 ± .004^{b}	92± 0.004^{e}	57.091± 0.003^{c}	45.100 ± 0.002^{a}
Ca	3094 ± 0.177^{c}	924± 0.109^{a}	10627± 0.845^{e}	3869 ± 0.254^{d}	2591 ± .042^{b}
Cd	0.400 ± 0.002^{c}	0.370± 0.002^{b}	2.120± 0.002^{e}	0.560 ± 0.001^{d}	BD
Co	3.780± 0.002^{c}	3.630± 0.003^{b}	5.590 ± 0.001^{e}	4.262 ± 0.004^{d}	3.481 ± 0.004^{a}
Cr	49.221± .009^{b}	62± 0.010^{d}	47.171± 0.006^{a}	82.731 ±0.007^{e}	55.581 ±0.003^{c}
Cu	43 ± 0.011^{d}	29.452± 0.008^{c}	96± 0.001^{e}	21.171 ±0.008^{a}	22.800 ±0.007^{b}
Fe	18614±0.283^{c}	18864±0.987^{e}	17038±0.512^{b}	18716±1.102^{d}	12547±1.395^{a}
K	984±0.047^{b}	1100±0.111^{c}	1156±0.076^{d}	1939±0.130^{e}	960±0.043^{a}
Mg	953±0.039^{a}	998±0.071^{b}	7582±0.190^{e}	1900± 0.081^{d}	1042± 0.035^{c}
Mn	215± 0.010^{d}	171± 0.013^{b}	254± 0.005^{e}	129± 0.006^{a}	182± 0.004^{c}
Na	684± 0.035^{c}	684± 0.009^{b}	1287± 0.078^{e}	808 ± 0.036^{d}	666±0.035^{a}
Ni	11.312±0.022^{d}	7.660±0.016^{a}	11.687±0.018^{e}	10.463±0.021^{c}	8.891±0.020^{b}
Pb	66±0.011^{d}	27.242±0.032^{a}	30.931±0.020^{b}	30.900±0.035^{b}	41.210±0.025^{c}
V	48±0.003^{c}	52.761±0.009^{e}	43.250±0.001^{b}	51.703±0.002^{d}	32.511±0.001^{a}
Zn	3052±0.102^{c}	1132±0.072^{a}	3970±0.090^{d}	4274±0.173^{e}	1677±0.040^{b}

Data expressed as mean ± standard error of mean three replicate concordant values. Data with different superscripts are significantly different at p < 0.05 OGS, STDS, OFAS, ILLS, MAFS are old garage soil, stadium soil, Ofa-bolanle soil, Ilesa soil, May fair soil

Table 2: Metal concentrations associated with MPs in different soil samples (Rainy season) (mgkg^{-1})

Metals	OGS	STD	OFS	ILSS	MAFS
Al	3070 ± 0.160^{d}	4710 ± 0.400^{e}	1100 ±0.012^{b}	930 ±0.003^{a}	1266 ± 0.092^{c}
Ba	35.830 ± 0.173^{c}	24.570 ± 0.011^{b}	BD	BD	15.671± 0.006^{a}
Ca	1975 ± 0.033^{e}	390 ± 0.033^{a}	781 ± 0.007^{c}	501 ± 0.006^{b}	1403 ± 0.064^{d}
Cd	0.871 ± 0.001^{d}	1.260 ± 0.006^{e}	0.182 ± 0.006^{a}	0.320 ± 0.006^{b}	0.611 ± 0.006^{c}
Co	BD	BD	2.381 ± 0.016^{a}	1.833 ± 0.016^{b}	BD
Cr	23.631 ±0.008^{d}	37.172 ±0.007^{e}	0.820 ± 0.131^{a}	1.460 ± 0.008^{b}	13.901 ±0.005^{c}
Cu	25.350 ± 0.011^{e}	16.571 ±0.003^{d}	0.244 ± 0.004^{a}	0.432 ± 0.007^{b}	11.460 ± 0.124^{c}
Fe	12132 ± 1.847^{c}	16367 ± 0.019^{e}	11770 ± 0.006^{b}	12152 ± 0.006^{d}	4762 ± 0.295^{a}
K	675 ± 0.053^{c}	1033 ± 0.360^{e}	709 ± 0.341^{d}	617 ± 0.127^{b}	306 ± 0.213^{a}
Mg	615 ± 0.016^{d}	805 ± 0.034^{e}	474 ± 0.003^{c}	441 ± 0.006^{a}	452 ± 0.003^{b}
Mn	128 ± 0.004^{a}	85 ± 0.006^{d}	42 ± 0.001^{b}	21 ± 0.001^{a}	74 ± 0.003^{c}
Na	1267 ± 0.056^{e}	849 ± 0.072^{d}	690 ± 0.358^{b}	781 ± 0.013^{c}	443 ± 0.556^{a}
Ni	5.330 ± 0.013^{d}	5.630 ± 0.105^{e}	3.352 ± 0.008^{c}	1.129 ± 0.014^{a}	2.741 ± 0.026^{b}
Pb	41.561 ± 0.005^{d}	24.020 ± 0.042^{b}	1.241 ± 0.029^{a}	BD	24.233 ± 0.019^{c}
V	19.422 ± 0.036^{d}	32.750 ± 0.116^{e}	11.232 ± 0.058^{c}	0.051 ± 0.003^{a}	7.720 ± 0.003^{b}
Zn	2422 ± 0.303^{c}	1048 ± 0.987^{a}	BD	BD	1342 ± 0.094^{b}

Data expressed as mean ± standard error of mean of three triplicate concordant values. Data with different superscripts are significantly different at p < 0.05 OGS, STDS, OFAS, ILLS, MAFS, BD are old garage soil, stadium soil, Ofa-bolanle soil, Ilesa soil, May fair soil, below detection

Table 3 shows the concentrations of metals associated with MPs in fish organs from Osun, Oora and Opa Rivers during the dry season. Fe dominated across all sites, peaking in Oora River (58,924 mg kg^{-1}), followed by Opa and Osun rivers respectively, reflecting its strong affinity for MP surfaces. Oora fish organs had higher levels of Cu, Cr and Mn, while Opa showed the highest Pb and Ni concentrations. Cd remained low but consistent across sites and Zn exhibited moderate levels with minimal variation The results highlight the role of MPs as vectors for metal accumulation in aquatic organisms. Elevated TMs are of concern due to their persistence and bioaccumulation [61]. Higher Cu, Cr, and Mn in Oora River and Pb and Ni in Opa River suggest localized anthropogenic inputs, consistent with recent studies [62, 63]. Table 4 presents the concentrations of metals associated with MPs in fish organs from Osun, Oora and Opa Rivers during the rainy season. Fe dominated across all samples, peaking in Oora (66,402 mg kg^{-1}), followed by Osun (62,497 mg kg^{-1}), while Opa lower concentration (8,251 mg kg^{-1}). Al was also elevated, particularly in Oora, whereas Ca was highest in Osun River, indicating notable mineral accumulation. Cd concentrations were high in Osun and Oora Rivers but considerably lower in Opa River. Cr and Cu were highest in Oora, while Pb peaked in Opa. Zn was higher in Osun River, with decreasing levels across Oora and Opa. Ni was detected in Osun and Oora Rivers but below detection in Opa River, while other elements (Mn, V, Mg, Na, K) varied by location. The elevated concentrations of Cd, Pb, and Cr raise serious

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Table 3: Metal concentrations associated with MPs in different fish samples (Dry season) (mgkg^{-1})

Metals	OSFT	ORAFT	OPAFT
Al	46.762 ± 0.013^{a}	187± 0.006^{b}	246.011± 0.007^{c}
Ba	24.940 ± 0.002^{c}	3.480 ± 0.001^{a}	20.710 ± 0.001^{b}
Ca	547± 0.093^{c}	573 ± 0.032^{a}	3275 ± 0.048^{b}
Cd	1.681 ± 0.003^{b}	2.751 0.001^{c}	1.660 ± 0.004^{a}
Cr	6.170 ± 0.003^{b}	33.661 ± 0.003^{c}	5.850 ± 0.004^{a}
Cu	1.930 ± 0.002^{a}	68.722 ± 0.004$_{c}$	6.191 ± 0.005^{b}
Fe	43718±0.605^{a}	58924±1.191^{c}	50152±1.802^{b}
K	589 ±0.043^{c}	303 ±0.021^{a}	408 ±0.005^{b}
Mg	65.100± 0.002^{a}	71.890± 0.006^{c}	67.460± 0.001^{b}
Mn	7.431± 0.001^{a}	22.701± 0.002^{c}	10.742± 0.001^{b}
Na	545 ±0.020^{c}	362 ±0.031^{a}	477 ±0.015^{b}
Ni	2.750±0.015^{a}	5.852±0.012^{b}	16.710±0.002^{c}
Pb	9.431±0.011^{a}	15.180±0.036^{b}	41.430±0.007^{c}
V	4.910±0.002^{a}	11.840±0.003^{c}	5.551±0.004^{b}
Zn	17.662±0.003^{c}	14.660±0.001^{b}	13.600±0.003^{a}

Data expressed as mean ± standard error of mean of three triplicate concordant values. Data with different superscripts are significantly different at p < 0.05. OST, OORAFT, OPAFT are Osun River fish tissue, Oora River fish tissue, Opa River fish tissue

Table 4: Metal concentrations associated with MPs in different fish samples (Rainy season) (mgkg^{-1})

Metals	OSFT	ORAFT	OPAFT
Al	54.332 ± 0.009^{b}	196.160 ± 0.031^{c}	24.360 ± 0.304^{a}
Ba	19.821 ± 0.006^{c}	1.833 ± 0.006^{b}	1.610 ± 0.006^{a}
Ca	5719 ± 0.449^{c}	246 ± 0.365^{a}	390 ± 0.107^{b}
Cd	2.60 ± 0.006^{c}	2.53 ± 0.006^{b}	0.77 ± 0.006^{a}
Cr	8.74 ± 0.003^{b}	13.61 ± 0.126^{c}	1.82 ± 0.103^{a}
Cu	5.03 ± 0.109^{b}	54.19 ± 0.006^{b}	1.44 ± 0.006^{c}
Fe	62497 ± 1.860^{b}	66402.3580^{b}	8251 ± 0.220^{c}
K	917.51 ± 0.043^{c}	319 ± 0.042^{b}	38.39 ± 0.044^{c}
Mg	123.94 ± 0.012^{c}	65.97 ± 0.005^{b}	19.43 ± 0.009^{a}
Mn	8.71 ± 0.006^{b}	9.77 ± 0.006^{c}	1.77 ± 0.006^{a}
Na	889 ±0.127^{c}	278 ± 0.057^{b}	51.89 ± 0.038^{a}
Ni	6.63 ± 0.024^{b}	3.34 ± 0.010^{a}	BD
Pb	4.08 ± 0.04^{b}	3.28 ± 0.009^{c}	6.32 ± 0.011^{c}
V	6.16 ± 0.006^{b}	7.78 ± 0.003^{c}	0.34 ± 0.006^{a}
Zn	32.51 ± 0.006^{c}	13.53 ± 0.006^{b}	1.11 ± 0.006^{a}

Data expressed as mean ± standard error of mean of three triplicate concordant values. Data with different superscripts are significantly different at p < 0.05. OST, OORAFT, OPAFT are Osun River fish tissue, Oora River fish tissue and Opa River fish tissue, respectively

ecological and health concerns due to their toxicity. (Mn, V, Mg, Na, K) varied by location. The elevated concentrations of Cd, Pb, and Cr raise serious ecological and health concerns due to their toxicity and bioaccumulation potential. Higher metal loads in the Oora and Osun Rivers likely reflect stronger anthropogenic inputs, including agricultural runoff and industrial discharges.

Ecological risk assessments

Table 5 shows the ecological risk of TMs in soil using the contamination factor (Cf) and the ecological risk factor (Erf). Zn showed the highest contamination (Cf = 40.302 dry; 13.76 rainy) and corresponding ecological risk (Erf = 40.30–13.76), indicating considerable to high risk. Cd also showed notable contamination (3.2–3.4) and high ecological risk (Erf = 97–103), classifying it as a considerable risk element. Pb showed moderate ecological risk, with Cf values of 1.457–3.143 and Erf ranging from 7.284 to 15.720. Other metals (Cu, Cr, Ni, Co, Mn, Fe, Al, Ba, and V) recorded low contamination (Cf < 1) and low ecological risk (Erf < 40), suggesting minimal ecological threat. These findings highlight Zn and Cd as major contributors to soil ecological risk, largely due to anthropogenic inputs such as fertilisers, industrial emissions and waste disposal [64]. Seasonal increases, particularly in the dry period, likely reflect reduced dilution and enhanced metal accumulation [65], highlighting the need for continuous monitoring and effective soil management strategies.

Table 6 summarizes the potential ecological risk of metals in fish organs using Cf and Erf across seasons. The results reveal pronounced element-specific and seasonal variations. Cd showed the highest risk, with

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Table 5: Ecological risk assessment (Soil)

Elements	Ave. conc (Dry Season) (mg/kg)	Ave. conc. (Rainy Season) (mg/kg)	Ref. Conc.	Tr	Cf (Dry season)	Cf (Rainy season)	ERF (Dry season)	ERF (Rainy season)
Al	8125	2215	82,300	2.500	0.099	0.027	0.247	0.067
Ba	64.360	15.210	425	1.500	0.151	0.036	0.226	0.054
Cd	0.690	0.650	0.2	30	3.446	3.242	103	97
Co	4.148	0.840	25	5	0.166	0.034	0.830	0.168
Cr	59.360	15.400	100	2	0.594	0.154	1.190	0.308
Cu	42.400	10.810	55	5	0.771	0.197	3.860	0.983
Fe	17,156	11,436	56,300	1	0.305	0.203	0.310	0.203
Mn	190	70.370	950	1	0.2	0.074	0.200	0.074
Ni	9.990	3.640	75	5	0.133	0.049	0.670	0.243
Pb	39.300	18.210	12.500	5	3.143	1.457	15.720	7.284
V	45.550	14.230	135	2	0.337	0.105	0.680	0.211
Zn	2821	963	70	1	40.302	13.76	40.300	13.76

CF, Ref. Conc., Tr, ERF and RI are contamination factor, Reference concentration, toxic response, ecological risk and risk index

Table 6: Potential ecological risk (Fish organs)

Elements	Ave metal conc. (Rain)	Ave metal conc. (Dry)	Ref. Conc.	Tr	Cf Rainy	Cf Dry	Erf (Rainy season)	Erf (Dry season)
Al	91.618	160.059	82,300	2.500	0.001	0.002	0.003	0.005
Ba	7.750	16.375	425	1.500	0.018	0.039	0.027	0.0585
Cd	1.966	2.029	0.200	30	9.831	10.143	295	304.290
Co	BD	0.247	25	5	BD	0.010	BD	0.050
Cr	8.060	15.228	100	2	0.081	0.152	0.161	0.304
Cu	20.220	25.615	55	5	0.368	0.466	1.838	2.330
Fe	45,596	50,931	56,300	1	0.810	0.905	0.81	0.905
Mn	6.750	13.626	950	1	0.007	0.014	0.007	0.014
Ni	3.320	8.435	75	5	0.040	0.113	0.222	0.565
Pb	4.560	22.015	12.500	5	0.370	1.761	1.823	8.805
V	4.760	7.433	135	2	0.040	0.055	0.0705	0.110
Zn	15.710	15.303	70	1	0.230	0.219	0.225	0.219

Cf, Ref. Conc., Tr, ERF and RI are contamination factor, reference concentration, toxic response, ecological risk and risk index

Cf values of 9.831 (rainy) and 10.143 (dry) and Erf values of 295 and 304.29, respectively, indicating a very high ecological risk (Erf ≥ 160) in both seasons. Lead (Pb) showed moderate risk, increasing in the dry season (Cf = 1.761; Erf = 8.805). In contrast, Cu, Ni, Cr, Zn, and Fe recorded low contamination (Cf < 1) and low ecological risk (Erf < 40). Despite its high concentration, Fe posed minimal risk due to its low toxic response factor. Similarly, Al, Mn, V and Ba showed negligible risk, while Co was below detection in the rainy season. These findings highlight Cd as the dominant ecological threat, likely from anthropogenic inputs.

Estimated dietary intake (EDI) of MPs in Fish organs

Table 7 showed the estimated dietary intake (EDI) of MPs in fish organs. The EDI values were between 9.70×10^{-2} and 3.08×10^{-1} with the highest intake in the Opa River (0.308), Osun (0.160) and Oora (0.097). On the other hand, the values of the dry season exposure were significantly high, ranging between 2.12×10^{-1} and 4.28×10^{-1} with the Osun River having the highest exposure (0.428), then Opa (0.279) and Oora (0.212). The increased dry-season EDI, especially in the Osun River, is possibly due to anthropogenic factors, such as wastewater discharge and reduced dilution capacity. A similar trend was observed by [66], in which MP concentrations in aquatic organisms were significantly higher during low-flow periods. The consumption of MPs poses serious health risks due to their ability to penetrate biological membranes, bioaccumulate in tissues, and induce cellular stress [55].

Table 7: Estimated dietary intake (EDI) of MPs in Fish organs

Sample	CM (g) (Rain)	CM (g) (Dry)	CR(Kg)	BW(Kg)	EDI (Rain)	EDI (Dry)
Osun fish organs	266	713	0.036	60	1.60E-01	4.28E-01
Oora fish organs	162	353	0.036	60	9.70E-02	2.12E-01
Opa fish organs	513	465	0.036	60	3.08E-01	2.79E-01

CM, CR, BW, and EDI are the mean MP abundance in fish, average daily fish consumption, body weight, and estimated dietary intake, respectively.

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Health risk assessment

The evaluation of the health risks associated with consuming fish contaminated with metals is presented in Table 8. In both seasons, the hazard quotient (HQ) values for Cr and Pb were over the safety limit (HQ > 1), with values of 2.10 to 3.14 for Cr and 1.11 to 2.59 for Pb, indicating that there were substantial non-carcinogenic dangers to consumers. According to [61] and [67], exposure to lead is associated with neurological, haematological, and renal abnormalities, whereas hexavalent chromium has been connected to oxidative stress and organ damage. Cd also showed a non-carcinogenic risk in the dry season (HQ = 2.22), but not in the rainy season (HQ=0.834). Long-term exposure to cadmium is associated with kidney dysfunction and bone demineralisation [68]. Other metals, Cu, Ni, V, Zn, Ba, Mn, and Al, had HQ values less than 1, indicating lower individual risk, but additive or synergistic effects remain a concern. The CR values of Cr (1.54×10^{-2} - 2.31×10^{-2}), Ni (2.88×10^{-2} - 3.41×10^{-2}), and Cd (1.55×10^{-2} - 4.12×10^{-2}) were much higher than acceptable values (10^{-6} - 10^{-4}), suggesting high lifetime cancer risk.

Table 8: Health risk assessment (Fish)

Metals	Average daily dose (ADD) (Rain) (mg/kg/day)	Average daily dose (ADD) (dry) (mg/kg/day	Hazard quotient (HQ) (Rain season)	Hazard quotient (HQ) (Dry season)	Carcinogenic risk (Rainy season)	Carcinogenic risk (Dry season)
Al	6.60E-02	1.06E-01	9.40E-03	1.52E-02		
Ba	6.74E-03	9.11E-03	3.37E-02	1.68E-02		
Cd	8.34E-04	2.22E-03	8.34E-01	2.22	1.55E-02	4.12E-02
Cr	6.29E-03	9.43E-03	2.1	3.14	1.54E-01	2.31E-01
Cu	1.06E-02	2.45E-02	2.64E-01	6.13E-01		
Mn	5.60E-03	7.83E-03	4.00E-02	5.60E-02		
Ni	3.47E-03	4.09E-03	1.70E-01	2.05E-01	2.88E-01	3.41E-01
Pb	9.05E-03	3.88E-03	2.59	1.11	4.40E-03	1.90E-03
V	3.05E-03	5.77E-03	3.39E-01	6.42E-01		
Zn	6.33E-03	1.91E-02	2.11E-02	6.36E-02		

MP types and chemical composition

MP types in soil and fish tissues were identified based on spectral characteristics and functional groups (Figure 8). Polyamides (PAs), polyethene terephthalates (PETs), polyethenes (LDPEs and HDPEs), polyvinyl chlorides (PVCs), polypropylenes (PPs), polystyrenes (PSs), and polycarbonates (PCs) are among the common plastics found.

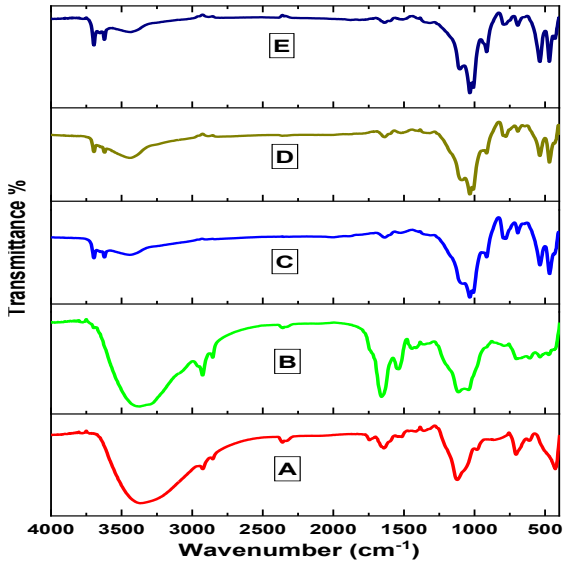

Fig. 8 FTIR spectra of MNPs found at old garage roundabout (A), Mayfair roundabout (B), Ilesa roundabout (C), stadium roundabout (D) and Ofa-Bolanle roundabout (E).

Conclusions

This study provides compelling evidence of the co-occurrence of microplastics (MPs) and toxic metals in soils and freshwater fish across urban centres in southwestern Nigeria, with marked spatial and seasonal variability. The elevated abundance of MPs during the rainy season, alongside higher metal concentrations in the dry season, stresses the critical role of hydrological processes in regulating contaminant distribution and dynamics. The dominance of fibres and fragments suggests predominantly secondary plastic sources, while the considerable accumulation of MPs in fish organs confirms both ingestion and systemic translocation. The strong association between MPs and toxic metals, particularly Cd, Pb, and Cr, highlights the role of plastics as effective vectors that enhance metal mobility, bioavailability, and potential toxicity. Particularly for cadmium and chromium, ecological and human health risk assessments showed moderate to extremely high risks. These results highlight the urgent need for better waste management techniques, more stringent pollution control methods, and environmental monitoring of urban soil and freshwater systems.

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