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INSIGHTS INTO MICROPLASTICS POLLUTION IN AQUATIC ECOSYSTEM: A SHORT REVIEW OF SAMPLING AND ANALYSIS METHODS

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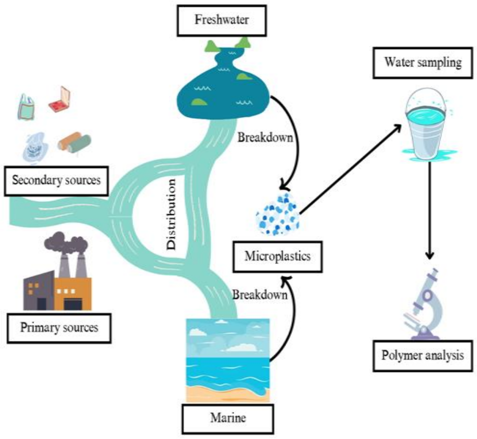
Graphical abstract



Abstract

The prevalence of microplastics in the environment and their potential adverse impacts on wildlife and human well-being make them a pressing global ecological issue. The objective of this review is to provide better understanding prevalence and distribution of microplastics, factors influencing their concentration and transport, the sampling technique and polymer identification analysis used in various studies and future research needs. The identification of microplastic polymer types is also explored, including techniques such as Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy. It also points out the many types of sampling equipment and materials, as well as sample preparation and analysis. Sampling method and polymer identification analysis of microplastics may vary on the circumstances of the location and question of the research. Suitable and standardize for both methods should be prioritized to ensure reliable and precise collection of data regarding the microplastic in river and estuarine.

Keywords: Microplastics, sampling, analysis, river, marine

Abstrak

Penyebaran mikroplastik dalam alam sekitar dan potensi kesan negatif terhadap hidupan liar dan kesejahteraan manusia menjadikannya isu ekologi global yang mendesak. Objektif kajian ini adalah untuk memberikan pemahaman yang lebih baik tentang kelaziman dan penyebaran mikroplastik, faktor yang mempengaruhi kepekatan dan pengangkutannya, teknik persampelan dan analisis pengenalpastian polimer yang digunakan dalam pelbagai kajian dan keperluan penyelidikan masa hadapan. Pengenalpastian jenis polimer mikroplastik juga diterokai, termasuk teknik seperti spektroskopi inframerah transformasi Fourier (FTIR) dan spektroskopi Raman. Ia juga menunjukkan pelbagai jenis peralatan dan bahan pensampelan, serta penyediaan dan analisis sampel. Kaedah pensampelan dan analisis pengenalpastian polimer mikroplastik mungkin berbeza mengikut keadaan lokasi dan objektif penyelidikan. Kesesuaian dan penyeragaman untuk kedua-dua kaedah harus diutamakan untuk memastikan pengumpulan data yang boleh dipercayai dan tepat mengenai mikroplastik di sungai dan muara.

Kata kunci: Mikroplastik, analisis, persampelan, sungai, marin

1.0 INTRODUCTION

Microplastics are small plastic particles measuring less than 5 mm in size [1] which can originate from a variety of sources such as cosmetic products, textiles, and industrial processes, and can enter the environment through various pathways, including wastewater discharge, littering, and erosion of plastic waste [1;2]. Anthropogenic activities such as commercial product development are the main contributor of the microplastics accumulation in the environment [3;4]. Microplastics pose a significant threat to the environment, as they are highly persistent and can accumulate in the food chain, potentially causing harm to both wildlife and humans [5] and become microplastics is one of the worlds rising concerns recently. Plastics uses globally keep on increasing each year also contribute to this troubling issue. In 2021, approximately 390.7 million metric ton/year of plastics produced globally. In that year, China leads the way by producing the highest as one third of world plastics production (66). There are six plastics groups significantly from the industry such as polyethylene (PE), polypropylene (PP), polyvinylchloride (PVC), polystyrene (PS), polyurethane (PUR) and polyethylene terephthalate (PET) [6]. Plastics uses in wide range of field either industry, agriculture, aquaculture, and land used. When plastics enter the environmental intentionally or unintentionally and exposed to the environment eventually will cause the plastic to breakdown in mechanical and psychochemical properties and resulting the plastics fragment formation called microplastics ([7]. Some studies also discovered that microplastics can be found in numerous species from human brains to blood and digestive system [3;5]

Microplastics can be found in numerous environmental niches such as fresh water, marine, soil and even air [8]. These ecosystems are important aquatic environments that provide vital ecosystem services and support numerous human activities [9]. However, these environments are also susceptible to microplastic pollution, as they receive inputs of plastic waste from upstream sources and can act as sinks for microplastics carried by ocean currents [10; 11]. Therefore, it is important to understand the sources and fate of microplastics in rivers and estuaries, as well as their ecological and health impacts. In the freshwater environment, microplastics are mainly caused by the disposal of plastic waste, effluent from domestic, industrial and wastewater treatment plant, shipping, fishing, aquaculture through water runoff then disperse through the environment [12]. The aim of this review paper is to provide a comprehensive overview of the sampling and analysis methods used to detect microplastics in aquatic environment. By synthesizing the current knowledge on microplastics in these environments, this review will provide insights into the state of the methods in sampling and analysis methods and highlight areas for future research. Figure 1 shows the summary of this review paper.

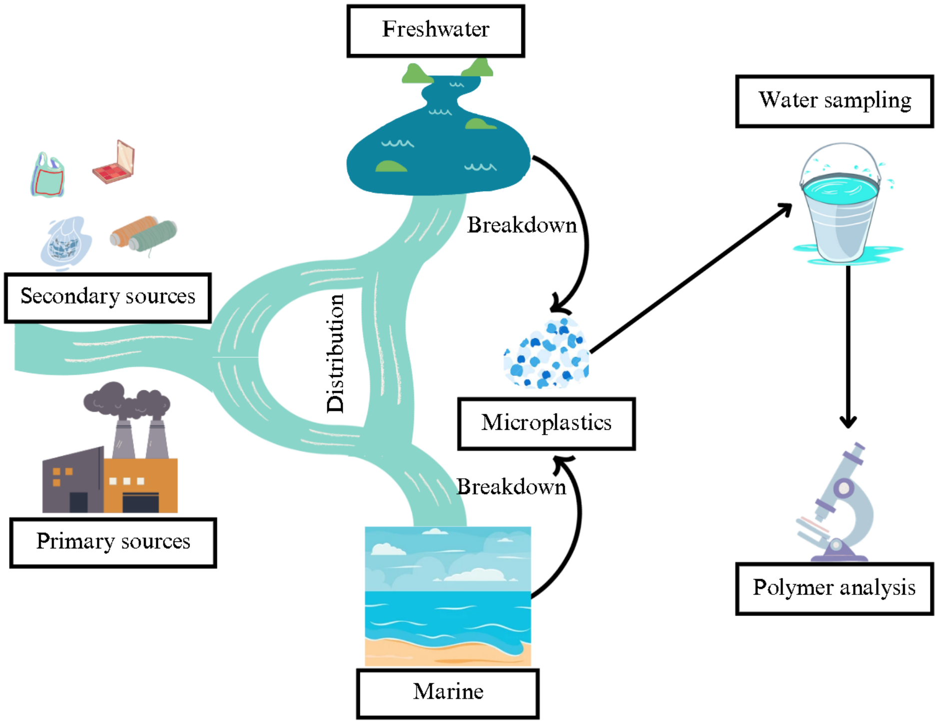


Figure 1 Summary of the Insights into Microplastics Pollution in Rivers and Estuaries: A Short Review of Sampling and Analysis Methods

The study has been conducted to discuss "Insights into Microplastics Pollution in Rivers and Estuaries" by searching for journals and articles using several electronic databases such as Science Direct, Scopus and Google Scholar. Boolean keyword combinations of "microplastics", "water sampling", "river and estuaries", and "analysis method" were used. 73 total references from both article paper and conference paper were used. When a publication was found, a systematic search was conducted inside the website of this specific journal to find relevant studies. Additional publications were then discovered in the reference lists of the resulting journal papers[13]. There is some inclusion and exclusion criteria for the chosen journals and articles based on the title and abstract such as i) paper published in the last decade, between 2010-2020 ii) topic associated with microplastics (water sampling analysis) iii) analysis method iv) English literature. Studies that are focusing on microplastic in marine were excluded in this study.

2.0 SHAPE OF MICROPLASTICS

The prevalence and distribution of microplastics in rivers and estuaries are a growing concern due to their potential ecological risk and human health impacts [14]. Several studies have reported microplastics in various rivers and estuaries around the world, with high concentrations found in urbanized areas and stormwater runoff. Despite of microplastics is remain transported to sediment by influence of wind, hydrodynamic condition, bioconcentration, the shapes of microplastics themselves, aggregation and biofouling, most of them are buoyant [15]. Microplastics, originating from both primary and secondary sources [16], result from the disintegration of larger plastics into nano-, micro-, and macro-sized fragments through weathering processes like wind abrasion, wave action, photodegradation, hydrolysis, and exposure to ultraviolet radiation from sunlight [17], before being released into the environment. The production of secondary microplastics is facilitated by the process of fragmentation, which involves three distinct stages of bio-fragmentation, assimilation, and bio-deterioration [18]. Interestingly, the ocean is not the initial destination for microplastics, as these particles originate from human activities on land and accumulate in rivers before being transported into the ocean by water flow [19]. This was noted in a recent study by Li *et al.* [20] highlighting the importance of understanding the sources and pathways of microplastic pollution in aquatic environments. In addition, it is important to note that fragmentation can have a significant impact on the properties and behaviours of microplastics, such as their buoyancy and potential for ingestion by aquatic organisms. This can further exacerbate the negative impacts of microplastic pollution on marine ecosystems, emphasizing the need for effective mitigation strategies. The factors that influence the concentration and transport of microplastics in rivers and estuaries include urbanization, industrial activities, wastewater treatment plants, and sediment transport.

The types of microplastics found in rivers and estuaries include fibers, beads, and fragments [20]. Fibers are the most common type of microplastic found in rivers and estuaries and are often derived from textiles and clothing. Beads are commonly used in personal care products and can be found in high concentrations in estuaries. Fragments are often the result of degradation of larger plastic items such as bags and packaging. Meanwhile, Figure 2 shows the six commons shape of microplastics found in river and estuarine based on the study from [21].

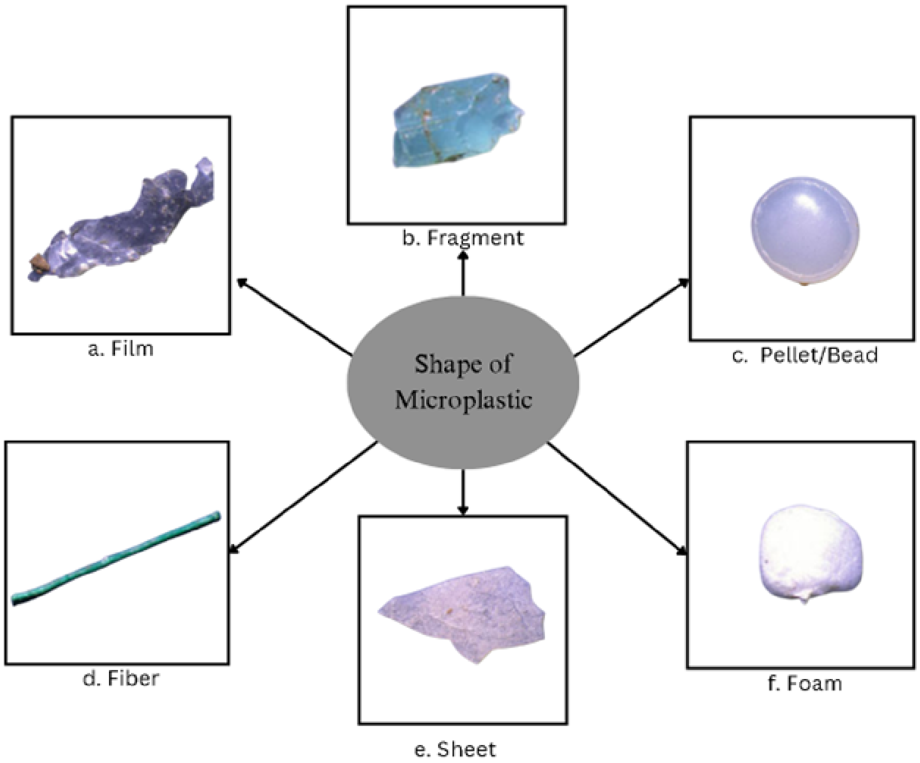


Figure 2 Shape of microplastics found in rivers and estuaries

According to Wu *et al*. [21], an intriguing finding suggests that the morphological characteristics of microplastic samples can offer insights into their potential sources. For instance, film-like microplastics are likely derived from bags or wrapping materials, while line/fiber-shaped ones are typically associated with fishing lines, clothing, or textiles. Interestingly, studies conducted in China's four estuaries, including the Three Gorges Reservoir, Taihu Lake, and freshwater areas of Wuhan, revealed a higher prevalence of fibers and granules in the microplastic samples. The observed diverse patterns in different study areas highlight substantial disparities in the sources of microplastics across various geographical locations. Notably, densely populated regions demonstrate a pronounced prevalence of fibers. In Southeast Asia, there is a growing trend of research focused on microplastics in numerous domains. Studies have explored the pathways of microplastic accumulation in soil as well as developed analytical methodologies for their analysis [22].

Table 1 provides a comprehensive summary of the shape of microplastics found in several rivers across Southeast Asia. The studies cited in the table offer valuable insights into the types of microplastics present in each location, shedding light on the extent of plastic pollution in these water bodies. Table 1 unveils an intriguing range of microplastic shapes in Southeast Asian rivers, encompassing microbeads, filaments, fragments, fibers, films, monofilaments, foam, pellets, and spherules. This diverse array reflects the multitude of sources and pathways through which microplastics infiltrate these waterways. Variances in shape are attributed to local activities, population density, waste management practices, and proximity to pollution sources. These findings underscore the urgent need for effective waste management and pollution control strategies in these highly affected regions, emphasizing the widespread presence of microplastics and the significance of addressing plastic pollution on a regional scale. By studying the types and shapes of microplastics, we gain valuable insights into their origins, transport mechanisms, and potential impacts on aquatic ecosystems, enhancing our ability to devise targeted solutions for this pervasive issue.

In Sarawak, Malaysia, the Kuap River, Maong River, and Sarawak River were found to contain microbeads, filaments, fragments, fibers, and films. According to studies on the microplastics at the Sarawak River, anthropogenic activity is the main factor determining the quantity of microplastics in the river, in addition to the river being adjacent to the place of trash dumping [23]. In Terengganu, Malaysia, the Dungun River was predominantly populated by fragments and fibers [24]. Yang Hwi *et al*. [24] conducted a study that examined the sampling locations along the Dungun River, highlighting the considerable impact of local activities on the ecosystem. The Dungun River, renowned as a prime fishing spot, holds significance due to its close proximity to the South China Sea. This study aimed to assess the increased anthropogenic influence resulting from local activities, making it a valuable investigation in understanding the ecological dynamics of the area. Additionally, due to the area's high population density and the possible impact of microplastics from anthropogenic activities, the Langat and Kelantan River were chosen as research sites. For instance, in the Selangor state of Malaysia, the Langat River basin is known to provide water to more than 2 million people in the area surrounding the river basin water catchment.

Table 1 Shape of Microplastic

Location	Shape of Microplastics	References
Kuap River, Maong River and Sarawak River, Sarawak	Microbead, filament, fragment, fiber, film	[25]
Dungun River, Terengganu	Fragment and fiber	[24]
Langat and Kelantan River	Fragment and fiber	[26]
Brantas River, Indonesia	Fragment, fiber, film, and pellet	[27]
Citarum River, Indonesia	Fragment, fiber, film, monofilament and foam	[28]
Surabaya River, Indonesia	Film, fragment, fiber, foam, and pellet	[29]
Ciwalengke River, Indonesia	Fragment and fiber	[30]
Chao Phraya River, Thailand	Fragments, pellets, films, and fiber.	[31]
Chi River Basin, Thailand	Fiber, fragment, pellet and foam	[32]
Tapi-Phumduang River and Bandon Bay, Thailand	Fibers, spherules, or fragments	[33]

3.0 TECHNIQUES FOR WATER SAMPLING TO IDENTIFY MICROPLASTICS

While the Environmental Protection Agency (EPA) of the United States and the Malaysian government have not yet developed specific sampling methods for microplastics in water, there has been significant progress in the field. Numerous studies have been conducted to develop and optimize techniques for sampling, separation, detection, and characterization of microplastics in water and wastewater bodies [34;35]. These efforts demonstrate a proactive approach to tackle the issue of microplastic pollution and pave the way for the development of effective sampling methods in the future. There are several methods for sampling water to detect microplastics, each with its own strengths and limitations.

Table 2 shows a variety of sampling apparatus and techniques that have been utilized in various locations to gather microplastic samples from rivers globally, with a predominant focus on Asia. A variety of apparatus and techniques have been employed to collect water samples for microplastic analysis in different locations worldwide. These methods include using stainless steel buckets and sieves, water samplers with membrane filters, neuston nets, nylon plankton nets, manta trawls, glass containers, pumps, plankton nets, and more. The sizes of the apparatus range from specific mesh sizes (e.g., 0.3-5mm) to varying diameters and lengths. Each method is tailored to suit the specific requirements of the sampling location. These diverse approaches highlight the adaptability and innovation in microplastic sampling methodologies across different regions, emphasizing the global effort to understand and combat microplastic pollution in water bodies.

Numerous methodologies have been devised to quantify microplastics in water, each offering unique advantages and limitations. This presentation aims to explore the diverse array of sampling techniques employed for assessing microplastic concentrations in aquatic environments. By delving into the distinct characteristics and considerations of these methods, we can gain a deeper understanding of their applicability, reliability, and potential implications for microplastic research and monitoring. As reported by Table 3, there are five types of microplastics water sampling: surface water sampling, vertical water column sampling, passive sampling, trawl sampling and pump sampling [29,32,36].

One common method is surface water sampling, which involves collecting water samples from the surface of a water body using a fine mesh net. Surface water sampling is a popular approach for identifying microplastics in bodies of water such as rivers, lakes, and seas. Water samples are collected from the surface of the water using a bucket or a bottle in this approach. To catch microplastics, collected water samples are filtered via a mesh or a membrane [37]. Generally, a bulk amount of water will be taken from the water surface before passing through either manta net or plankton net to filter unnecessary substance before being transferred to glass container. Instead of stationary sampling at certain point, microplastics sampling can also be done by towing either manta trawl or plankton net using a boat from certain point to another. The boat will sail contrary of the water current in certain amount of time or distance depend on the suitability of the research. Commonly during manta towing, flow meter will be utilized to calculate the total volume of sampling of water. It is highly recommended to put the manta trawl beside the boat instead of behind the boat because of water turbulence caused by the boat propeller may affect the amount of microplastic captured. There are various advantages to sampling surface water. It is simple and affordable to carry out, making it a viable option for large-scale sampling programmes. It also enables the collecting of water samples from various sites inside the body of water, which can offer spatial information regarding the distribution of microplastics. Moreover, surface water sampling can collect both floating and suspended microplastics, allowing for a more complete evaluation of microplastic contamination in the water body [36; 38].

However, surface water monitoring for microplastics has certain limitations. This method may not capture all microplastics present in the water as some particles can sink or float, leading to incomplete detection. Additionally, the recovery rate of microplastics using filters can be poor, meaning that not all particles will be captured in the water sample. The choice of filter material and particle size can also influence the outcomes, as smaller pore sizes have the potential to capture more microplastics but may experience faster clogging. In order to overcome these limitations, an alternative technique known as vertical water column sampling can be employed. The researchers, Lestari *et al*. [29], employed a modified manta trawl with varying layered depths to capture microplastics. This approach allowed for simultaneous sample collection at different water levels. The microplastics sampling conducted in Surabaya River revealed that the middle depth of the river exhibited a high abundance of microplastics. This method involves sampling water at various depths, providing a more comprehensive assessment of microplastic distribution throughout the water column. Vertical water column sampling is a technique for collecting water samples at various levels in the water column in order to examine the vertical distribution of microplastics [38]. This approach can reveal the origins of microplastics and how they migrate through the water column, typically using tools like Niskin bottles or plankton nets [39].

These samples are then processed by passing them through mesh or membrane filters to capture microplastics. The collected microplastics are subsequently examined and analyzed using microscopy or spectroscopic methods. This approach offers several advantages in assessing microplastic distribution. By sampling throughout the water column, it provides a more comprehensive understanding of the vertical distribution patterns of microplastics. This information is crucial for identifying potential sources and pathways of microplastics within the aquatic environment. Such insights gained from vertical water column sampling contribute to a more comprehensive assessment of the impact of microplastics on marine ecosystems and can inform effective mitigation strategies [40]. It can also detect microplastics that are not present in surface water samples, such as those that have sunk to the bottom of a body of water or are floating at greater depths[40; 41; 42]. However, vertical water column sampling also comes with certain limitations. It is a more time-consuming and costly method compared to surface water monitoring, as it requires specialized equipment and more complex sampling techniques.

Table 2 Sampling Techniques for Microplastics in Rivers Worldwide

Location	Apparatus	Size	References
Baram River, Sarawak	Stainless steel bucket Stainless steel sieve	- 0.3-5mm(mesh)	[43]
Kuap River, Maong River and Sarawak River, Sarawak	Water sampler Membrane filter	4.2L 0.45µm pore size	[25]
Dungun River, Terengganu	Stainless steel bucket Stainless steel sieve	36cm(diameter)x34cm(height) 200mm(diameter),60 µm(mesh)	[24]
Langat River and Kelantan River	Neuston net	300µm(mesh)	[26]
Cherating River, Pahang	Nylon plankton net	100µm(mesh),0.3 in(diameter),1m(length)	[44]
Brantas River, Indonesia	Stainless steel sieve	0.3mm and 5mm	[27]
Citarum River, Indonesia	Manta trawl	125µm(mesh),	[28]
Surabaya river, Indonesia	Manta trawl	333µm(mesh)	[29]
Ciwalengke River, Indonesia	Glass container	1 litre	[30]
Chao Phraya River, Thailand	Manta trawl	300µm(mesh)	[31]
Chi River Basin, Thailand	Pump Stainless steel sieve	- 45µm(mesh),	[32]
Tapi-Phumduang River and Bandon Bay, Thailand	Containers	5 litres	[33]
29 rivers in Japan	Plankton net	335µm(mesh)	[45]
Zhangjiang River, China	Steel Bucket Manta net	20 litres 330µm(mesh),	[46]
Pearl River, China	Manta trawl	330µm(mesh)	[48]
Qing River, China	Stainless steel bucket Stainless-steel screen mesh	- 5mm	[49]
Haihe River, China	Manta trawl	333µm(mesh)	[50]
Han River, South Korea	Manta net Jet pump	100µm(mesh)	[51]
Nakdong River, South Korea	Stainless steel beaker Portable net	20cm(diameter) 20 mm (mesh)	[52]
Saigon River, Vietnam	Bucket (fibers analysis) Plankton net	- 300µm(mesh)	[53]
Fengshan River, Taiwan	Hemp sling and steel bucket Sieve	- 50 mm, 297 mm and 5000 mm	[54]
Parnaiba River and Jaguaribe River, Brazil	Plankton net	120 and 300µm(mesh)	[55]
Antua River, Portugal	Motor water pump	0.055 mm(mesh)	[56]
Esmeraldas River, Ecuador	Amber glass bottle Sieve	500 ml	[57]

Additionally, factors such as turbulence and stratification within the water column can affect the distribution of microplastics, potentially impacting the accuracy of the results. Despite these challenges, vertical water column sampling remains a valuable tool for investigating the vertical distribution of microplastics and gaining insights into their sources and transport within aquatic ecosystems.

Passive sampling is a more recent method that involves the utilization of specialized filters capable of being left in the water for an extended duration to collect microplastics. In the study conducted by Kataoka *et al*. [45], microplastics were collected by suspending a net with a mesh size of 335 µm from a bridge over the river. The collection period ranged from 5 to 30 minutes. Passive sampling offers the advantage of ensuring the safety of the sampler during the collection process, allowing sampling to be conducted regardless of the water flow conditions. In spite of the fact, it is important for the sampler to monitor the water level tide, as it may fluctuate over time, in order to ensure that water passes through the net evenly. Passive sampling is a technique for collecting microplastics in bodies of water that does not need actively collecting water samples. Passive samplers are put in the water for a set amount of time, enabling microplastics to build on the sampler's surface. Passive sampling offers several advantages overactive sampling methods. One key advantage is its ability to provide a more accurate assessment of organisms' exposure to microplastics. By allowing microplastics to accumulate on the sampler surface over an extended period, passive sampling captures a wider range of particles and provides a more comprehensive representation of microplastic levels in the environment. This longer exposure time also contributes to a higher recovery rate compared to certain active sampling approaches. As a result, passive sampling is a valuable technique for obtaining reliable data on the presence and abundance of microplastics in aquatic ecosystems (45). Passive sampling methods offer flexibility in capturing microplastics through the use of different materials such as open or mesh bags, films, and sorbent materials. Each material has unique characteristics that can influence the types of microplastics collected, including their size and shape. This diversity in sampler materials allows for a more comprehensive understanding of the microplastic composition in water samples [58]. Though, it is important to acknowledge the limitations of passive sampling. Not all microplastics present in the water may adhere to the sampler surface, potentially resulting in an underestimation of the true microplastic load. The selection of sampler material and configuration is critical, as it can impact the efficiency of microplastic collection. Some materials may be more effective at capturing microplastics than others, leading to variations in results depending on the chosen sampler. Careful consideration and standardization of sampling protocols are necessary to minimize these limitations and ensure accurate assessment of microplastic pollution in aquatic environments.

An alternative method for water sample for microplastics collection is pump sampling. A pump is used in this approach to pull water through a filter, catching microplastics in the water sample [38; 59]. Microscopy or spectroscopic methods are then used to identify and quantify the microplastics in the filter. Pump sampling provides various benefits over other techniques of water sampling. It is quite simple to operate and can gather water samples from a wide range of water sources, including rivers, lakes, and seas. It also has a high recovery rate since the filter captures the majority of microplastics in the water [60]. Furthermore, passive sampling enables precise measurement of water volume, as it allows for specific control and measurement of the water flow rate. This precise control facilitates accurate determination of the volume of water being tested. Pump sampling, however, has several limits which are the size and porosity of the filter can have an impact on the sorts of microplastics collected. It is also affected by elements such as water flow rate and turbulence, which alter the dispersion of microplastics in the water.

Table 3 shows overview of each sampling technique has its own advantages and disadvantages. The selection of water sampling methods for microplastics in rivers and estuaries is contingent upon research objectives and resource availability, as noted by Pasquier *et al*. [61].

Table 3 Advantages and Disadvantages based on each technique of sampling

Technique of Sampling	**Advantage**	**Disadvantage**
Surface water sampling	-Easy to collect samples from water surfaces. -Representative of water's top layer. -Useful for studying floating microplastics. -Cost-effective and simple to implement.	-May miss microplastics present at lower depths. -Susceptible to contamination from air and surrounding environment.
Vertical water column sampling	-Captures microplastics at various depths. -Provides insight into vertical distribution.	-Requires specialized equipment for vertical profiling. -Possibility of contamination during deployment and retrieval.
Trawl Sampling	-Efficient for collecting larger debris. -Suitable for open water and rivers. -Can cover large areas quickly.	-May not capture smaller microplastics. -Disruption of benthic habitats during trawling may impact the ecosystem.
Pump Sampling	-Efficient for collecting water from depths. -Allows for continuous sampling. -Useful for real-time monitoring.	-Risk of equipment-induced contamination. -Potential disturbance of water column during pumping.
Passive Sampling	-Accumulates microplastics over time. -Can be deployed in various water environments. -Low disturbance to the water column.	-Limited to specific types of microplastics. -May not provide real-time data. -Requires retrieval for analysis.

Surface water sampling and sediment sampling are commonly employed techniques, each offering unique advantages and limitations. Additionally, passive sampling holds promise for long-term monitoring purposes. Nevertheless, regardless of the chosen approach, ensuring accurate and representative sampling is crucial for comprehending the composition and abundance of microplastics in these aquatic environments. Ultimately, the selection of a sampling method should align with the research goals and available resources, emphasizing the importance of a well-informed decision-making process.

4.0 POLYMER IDENTIFICATION ANALYSIS OF MICROPLASTICS IN WATER SAMPLES

Polymer identification analysis of water samples for microplastics is the process of identifying the types of microplastic particles present in a water sample. Several previous studies have utilized the guidelines provided by the National Oceanic and Atmospheric Administration (NOAA) as a reference and have adapted their methods to assess the abundance of microplastics in water samples. By modifying the NOAA guidelines, researchers were able to tailor their approaches to effectively measure microplastic levels in water samples. The identified polymers can provide information about the potential sources of the microplastics. One common method is Fourier transform infrared (FTIR) spectroscopy, which identifies the specific polymers in microplastic particles by analyzing their infrared spectra. Another method is Raman spectroscopy, which also analyzes the spectra of microplastic particles but uses laser light instead of infrared radiation. A newer method is pyrolysis gas chromatography mass spectrometry (Py-GCMS), which breaks down the microplastic particles into their individual components and identifies them using gas chromatography and mass spectrometry [2]. Table 4 shows that the analysis with different pre-treatment and approaches. Each of the method was chosen based on the as accessibility, location and targeted type of polymer to be find. In this table, various water sampling locations and their corresponding pre-treatment methods, analytical techniques, particle sizes, and types of polymers analyzed for microplastics are presented.

Table 4 Microplastic Analysis in Water Samples from Global Rivers: Pre-treatment, Analytical Methods, Particle Size, and Polymer Identification

Location	Pre-treatment	Analytical Method	Particle Size	Type of Polymer	References
Baram River, Sarawak	Fe (II), H_2O_2, NaCl	Stereomicroscope ATR-FTIR	0.3mm-5mm	PE, PET, PS, fibers, silicon polymer, nitrile	[43]
Kuap River, Maong River and Sarawak River, Sarawak	H_2O_2, FeH14O11S	Stereomicroscope 40X, ATR-FTIR	63-500µm.	EVA, PA, PP, PMMA, PS	[25]
Dungun River, Terengganu	H_2O_2	Stereomicroscope, FTIR	0.3-24.7µm	PP, PAN, RY	[24]
Langat River and Kelantan River	H_2O_2, $FeSO_4$	Stereomicroscope, Microscope, Spectroscopy, FTIR, Py-GCMS	<300µm–5000µm	PE, PP, PET, RY, PTFE	[26]
Cherating River, Pahang	Alcohol solution	Binocular dissection microscope	0.5-1.0mm	-	[44]
Brantas River, Indonesia	H_2O_2	FTIR	133 particles/m^3 - 5467 particles/m^3	PE, PVC, PC	[27]
Citarum River, Indonesia	H_2O_2, Fe	FTIR	0.06 particles/m^3	PE, PP	[28]
Surabaya river, Indonesia	H_2O_2, Fe, NaCl	Stereomicroscope, FTIR	1–5 mm	PE, PP	[29]
Ciwalengke River, Indonesia	-	Light binocular microscope 10X, Spectroscopy	(50-100 mm)	PS, PA	[30]
Chao Phraya River, Thailand	Fe (II), H_2O_2, NaI	Optical microscope, FTIR	0.05-0.3mm	PP	[31]
Chi River Basin, Thailand	H_2O_2,	ATR-FTIR	51-5000µm	PVC, PP, PE, AF PS	[32]
Tapi-Phumduang River and Bandon Bay, Thailand	-	FTIR	<1 mm	PP or PE, PET, and nylon.	[33]
29 rivers in Japan	NaCl,	Stereoscopic microscope, FTIR	<1 mm	PP, PS, PE	[34]
Zhangjiang River, China	H_2O_2, $FeSO_4$, NaCl	Stereoscopic microscope, micro-Raman spectroscopy	0.3-5.0mm	PP, PE	[46]
Pearl River, China	H_2O_2:FeSO4	micro-Raman spectroscopy	0.01-5mm	PP, PS, PE	[48]
Qing River, China	H_2O_2, $FeSO_4$	FTIR	>50µm	PE, PP, EPR	[49]
Haihe River, China	H_2O_2, NaCl	Stereomicroscope, m-FT-IR, SEM-EDS	500-2000µm	PE, EPC, PA, PP, PS, PU, cellulose, PET	[50]

Location	Pre-treatment	Analytical Method	Particle Size	Type of Polymer	References
Han River, South Korea	H_2O_2	FTIR	0.1-5mm	PE, PS, PTFE, PET, silicone	[44]
Nakdong River, South Korea	H2O2 and Fe (II)	FTIR	<300 mm	PP, PES, PE, PA, PS, PU, PVC	[51]
Saigon River, Vietnam	Protease and amylase, ZnCl2, H_2O_2	Stereomicroscope, FTIR,	50-1000μm	PE, PP, PET	[53]
Fengshan River, Taiwan	ZnCl2, H_2O_2	ATR-FTIR	50-297μm	PE, PET, PA, PES	[54]
Antua River, Portugal	ZnCl2, H_2O_2, Fe (II)	Stereomicroscope, ATR-FTIR	<5 mm	PE, PP, PS, PET, PVA	[56]
Esmeraldas River, Ecuador	H_2O_2, NaCl	Stereomicroscope 20×	-	-	[57]

Table 4 presents a comprehensive overview of microplastic analysis conducted on water samples collected from diverse locations worldwide, with a notable focus on Asian regions. The table provides valuable insights into the distribution and characterization of microplastics in different aquatic environments. Different pre-treatment methods and analytical techniques were employed to examine the particle size and types of polymers present. For example, in the Baram River, Sarawak, microplastics ranging from 0.3mm to 5mm were identified, including polymers such as PE, PET, PS, fibers, silicon polymer, and nitrile [43]. Other locations, such as the Dungun River in Terengganu and the Langat River and Kelantan River, utilized different methods to identify microplastics of varying sizes and polymer types. Each study contributes valuable insights into the global presence and characteristics of microplastics in different rivers, highlighting the need for comprehensive monitoring and mitigation strategies. The analysis of microplastics in water comprises multiple phases, including sample collection, preparation, and analysis.

The pre-treatment stage is an essential aspect of the sample preparation procedure because it removes any interfering compounds from the water sample and concentrates the microplastics for analysis. This process involves the removal of contaminants, such as organic matter or minerals, commonly found alongside microplastics in environmental samples. By eliminating these extraneous substances, pretreatment enhances the specificity of the analysis, allowing for a more precise measurement of the actual concentration of microplastics. Additionally, pretreatment minimizes interferences from various substances present in the samples, further contributing to the reliability and validity of the analytical findings. In essence, the pretreatment step is essential in refining the analysis to focus specifically on microplastics, thereby improving the overall accuracy of the study. Filtration is commonly employed in the pre-treatment process to remove bigger particles and debris from the water sample [38]. The filtered water is subsequently concentrated using sedimentation, centrifugation, or filtering via a lower particle size filter [62;63]. After that, the concentrated sample is suitable for microplastics examination. Many factors can have an impact on the pre-treatment procedure and the accuracy of microplastics water analysis. The size and kind of filter used are critical in capturing microplastics of interest while limiting the loss of smaller microplastics. The filtering process and flow rate can also influence microplastics collection efficiency and the risk of false positives or negatives

Additional pre-treatment procedures that may be used to increase the accuracy of microplastics water analysis include digestion, which involves breaking down organic content in the water sample using enzymes or oxidising agents [64]. This approach is effective for eliminating interfering organic waste and enhancing the recovery of microplastics. The common pre-treatment for microplastic by adding chemical substance digestion followed by the density separation. Type of microplastic that can be found after pre-treatment may vary depend on the chemical substance used. Most common pre-treatment digestion is Wet Peroxide Oxidation (WPO). Wet Peroxide Oxidation consist of iron sulfate solution, hydrogen peroxide and salt. Hydrogen peroxide was used to oxidize natural organic matter. Meanwhile, iron sulfate solution was used to catalyze the reaction. Salt was added to increase the density of the aqueous solution after natural organic material cannot be seen. Eventually density separation was applied using sieve or glass filter paper. There is another method known as Fenton Reaction using combination of hydrogen peroxide and ferrous sulfate. Most pre-treatment uses hydrogen peroxide as it cruciality in oxidization of organic matter. For that reason, several research just uses hydrogen peroxide as their main chemical for organic matter digestion without adding other substances. When the pre-treatment procedure is completed, the microplastics in the water sample can be evaluated using microscopy, spectroscopy, or chromatography methods. Each of these procedures has advantages and limits, and the methodology used will be determined by criteria such as the size and type of microplastics in the sample, the precision required, and the resources available.

In the realm of microplastics water analysis, microscopy methods play a pivotal role in visually identifying and quantifying microplastics. A diverse array of microscopy techniques is employed to investigate microplastics, encompassing stereomicroscopy, fluorescence microscopy, and electron microscopy, among others [38]. These advanced microscopy techniques facilitate the detailed examination and characterization of microplastic particles, enabling researchers to gain deeper insights into their presence and abundance in water samples. Moreover, hot needle test also can be conducted under optical microscopy to verify the microplastic compound since its reaction due to high temperature of the needle. Stereomicroscopy, often known as a dissecting microscope, is a method that produces high-resolution pictures of a material at low magnification (up to 100x). This approach is frequently used to identify and count bigger microplastics (those larger than 500 m). The fragments were handled or moved around with tweezers under a stereomicroscope to validate the static electricity property of the plastic particles. If the materials disintegrated or were easily destroyed it cannot be identified as plastic compound. If the particles retained their form, they were counted compound. Microplastics' form, colour, and texture may also be estimated via stereomicroscopy. Stereomicroscopes include a camera and a software package for automated counting and analysis of microplastics. The method of electron microscopy (EM) delivers great magnification (up to 1,000,000x) and high-quality pictures of the material. This approach is frequently used to examine the surface structure of microplastics in more depth. In microplastics investigation, two forms of EM are typically used: scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [12]. The exterior structure of microplastics is visualised using SEM, whereas the inside structure of microplastics is visualised using TEM [65]. Microscopy methods, in addition to ocular identification and counting, can be used to determine the size, shape, and colour of microplastics. The accuracy and precision of microscopy techniques are determined on the analyst's expertise and experience, as well as the quality of the equipment employed [64]. It should be highlighted that visual identification of microplastics is susceptible to misidentification. Some microplastics may resemble natural particles such as fibres or mineral grains. Without specialised analytical procedures, distinguishing between natural and manufactured particles can be challenging, leading to misidentification.

In microplastics water analysis, spectroscopy methods are frequently employed to identify and characterise microplastics based on their chemical composition know. Fourier-transform infrared spectroscopy (FTIR) and Raman spectroscopy are two types of spectroscopy methods extensively utilised in microplastics examination. The absorption or transmission of infrared light by a sample is measured using FTIR spectroscopy. Infrared radiation causes the molecules in the sample to vibrate, and the resultant spectra may be used to determine the sample's chemical makeup. FTIR spectroscopy is frequently used in microplastics water analysis to detect the types of polymers contained in the microplastics. The approach entails obtaining a water sample, filtering it to extract the microplastics, and then analysing the microplastics using FTIR spectroscopy. Meanwhile, Raman spectroscopy is a method for measuring the scattering of laser light by a material. The dispersed light may be utilised to detect the chemical content and structure of the material. Raman spectroscopy is frequently used in microplastics water analysis to detect the types of polymers present. The approach entails taking a water sample, filtering it to extract the microplastics, and then analysing the microplastics on the filter using Raman spectroscopy. FTIR and Raman spectroscopy are both non-destructive procedures, which means that the material is neither transformed nor destroyed during the study. These approaches can also offer information about the microplastics' size, shape, and colour. Nevertheless, the sensitivity of the instrument and the presence of interfering compounds in the sample restrict spectroscopic approaches [38].

Chromatography methods are often applied in microplastics water analysis to separate and identify distinct chemical components in a sample. Two types of chromatography techniques that are widely employed in microplastics analysis are gas chromatography (GC) and liquid chromatography (LC) [25]. Gas chromatography is a method of separating chemicals based on their volatility. The sample is vaporised before being transported through a stationary phase-packed column, which separates the various components depending on their interactions with the stationary phase [55]. In microplastics water analysis, chromatography methods such as gas chromatography (GC) and liquid chromatography (LC) are often employed to detect and quantify chemical substances related with microplastics. These compounds may include plasticizers, additives, and other pollutants that can be released into the environment by microplastics. GC and LC are strong procedures for isolating and identifying specific chemical components in a sample, and they can give useful information about the organic molecules linked with microplastics. The process of gas chromatography (GC) isolates a mixture of substances depending on their volatility. The sample is vaporised before being transported through a stationary phase-packed column, which separates the various components depending on their interactions with the stationary phase [11].

The accessibility and cost-effectiveness of different Polymer Identification Analysis methods for water samples vary. Fourier transform infrared (FTIR) spectroscopy is a common and relatively accessible method but may not be able to detect all types of polymers [1]. Raman spectroscopy is more expensive per analysis, more resource-intensive, and more specialised [66]. Pyrolysis gas chromatography mass spectrometry (Py-GCMS) is a newer method and requires specialized equipment and expertise, making it more expensive [67]. The selection of an appropriate method for microplastics analysis is contingent upon the specific research objectives and the available resources. While factors such as cost and accessibility hold significance, the paramount consideration in method selection should be the attainment of accurate and reliable results. Modification of analysis method by combining them together can lead to accurate interpretation and better understanding of microplastics. Striking a balance between practicality and data quality is crucial in order to ensure the robustness of the findings and the meaningful interpretation of the outcomes.

5.0 CONCLUSSION AND RECOMMENDATION

This review has provided an overview of microplastics sampling techniques and analysis methods in river water surface and discussed numerous methods used in various regions across the world. Existing data are often unmatchable because of diverse approaches used. For better understanding of microplastics distribution and contamination globally, procedures and methodologies should be standardized so that they are systematically along the analysis process. Among the limitations identified in current methods and technologies, several recommendations have been proposed to improve the study and management of microplastics. Firstly, implementing interlaboratory proficiency testing programs would help evaluate the accuracy and precision of microplastics analysis across different laboratories, thereby enhancing data quality confidence. Secondly, exploring and adopting emerging technologies such as machine learning, spectroscopy, and hyperspectral imaging could significantly improve the efficiency and accuracy of microplastics identification and quantification.

Additionally, combining multiple sampling techniques and analysis methodologies would allow researchers to capture a broader range of microplastic types, sizes, and locations, resulting in a more comprehensive understanding of microplastics pollution. Extending research efforts to include nanosized plastics is also crucial, as these present unique challenges and potential environmental implications compared to larger microplastics. Furthermore, developing and deploying specialized sampling instruments that target specific ecosystems, such as wastewater treatment facilities, stormwater runoff, and sediments, would enhance the accuracy of microplastics assessments.

Conducting more extensive and long-term monitoring programs is necessary to evaluate the trends and distribution of microplastics in rivers and estuaries over time. It is imperative for policymakers and environmental managers to recognize the significant threat posed by microplastic pollution. This approach should include the implementation of regulations and policies to reduce the production and consumption of single-use plastics and improve waste management practices.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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