JOURNAL OF
HYDRO-METEOROLOGY

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Research Article

Analytical methods used in microplastics identification: A review

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Received: 12 March 2024; Accepted: 08 April 2024; Published: 25 June 2024

Abstract: Microplastic research plays a crucial role in identifying microplastic polymers. Scientists use different methods such as Flame tests, Differential thermal scanning, Thermogravimetric analysis, and Infrared spectroscopy to accomplish this. The objective of this study incluce: (i) Firstly, it aims to summarize recent research trends on techniques for determining polymer types in various environments. It provides an overview of each technique and compares their strengths and limitations. (ii) Secondly, it determines the types of microplastics in surface water samples in the Saigon - Dong Nai River basin, during the period 2023. The Fourier transform infrared spectroscopy (FTIR) technique is applied according to the total attenuation method (ATR-FTIR). The study shows that it is possible to quantify and classify microplastics by manual observation or through observation or microscopy. However, determining the type of polymer is almost impossible. To overcome this limitation, scientists use a combination of physical (e.g., light microscopy, magnifying microscopy), chemical (e.g., spectroscopy), and thermal analysis techniques. The study results reveal that there are more than 60 types of microplastics present in the main water supply for daily drinking and drinking purposes of the people of Ho Chi Minh City and neighboring provinces. It provides a foundation for river basin water resource managers to propose appropriate water resource management measures and programs during the process of water exploitation and use in the area.

Keywords: Identification of microplastics; Microplastic analysis methods; Microplastics in surface water; Microplastics; Saigon River - Dong Nai.

1. Introduction

Since 2019, almost the whole world has been and will be struggling with a global pandemic - the Covid 19 epidemic. The World Health Organization has requested a 40% increase in disposable PPE (Polyphenylene Ether) production. Currently, the most commonly produced plastics are polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC), polyethylene terephthalate (PET) and polystyrene (PS) [1]. The term microplastics was first mentioned by Thompson and colleagues in 2004 [2]. Microplastics are defined as particles ranging in size from 1-5000 μm [3]. More clearly, it is distinguished as plastic particles, pieces, and fibers with a size of 5 to 1 mm, while a size of 1 mm or less is considered microplastic (nano-sized). They pose a potential risk to human health and the natural environment [4].

Identifying microplastics is quite complicated, there are many different methods [5]. Over the years, identification techniques such as Fourier transform infrared spectroscopy [6], Raman microscopy [7], gas chromatography desorption-mass spectrometry [8] or pyrolysis gas chromatography [9] have had many improvements and more in-depth research. Many studies have been published on methods to identify microplastics [5, 10–11]. Additionally, research publications [12, 13] summarize knowledge gaps and future research priorities. Looking at the total number of research publications on microplastics over the past and present years clearly shows that there is a need to strengthen technical methods for identifying microplastics beyond sampling.

New analytical tools need to be developed and integrated with existing instruments to tackle the challenges in the field of microplastic identification. The main issue to be addressed is the limited ability of current techniques to detect microplastics of different sizes. The detectable size limit of microplastics using current methods is only a few micrometers. However, there is a growing need to identify the presence, distribution, and polymer type of microplastics of various sizes, particularly in the nanoscale range. The detection and identification of polymers in nano-microplastics pose a significant challenge in microplastics research.

Accordingly, the purpose of this article is to present and compare current advanced techniques that can identify microplastics in water samples, thereby gaining a more general overview of the advantages and disadvantages of these methods. Techniques for identifying microplastics. In addition, the application of infrared techniques to identify microplastics in surface water samples of the Saigon - Dong Nai River was researched to find polymer types present in reality. There is an inference about their origin. Research results are the basis for researchers to consider choosing appropriate methods in research.

2. Materials and methods

2.1. Research process and structure diagram

The research process and structure diagram are shown in Figure 1. Figure 1 provides an overview of the study implementation process. We began by collecting analytical methods to identify microplastics and parameters from machinery and equipment manufacturers. We then synthesized, compared, and presented an overview and comparison of the strengths and limitations of each technique. After careful consideration, we chose a total attenuation Fourier transform infrared spectroscopy (ATR-FTIR) technique to study the polymers of microplastics in water samples of the river basin of Saigon - Dong Nai.

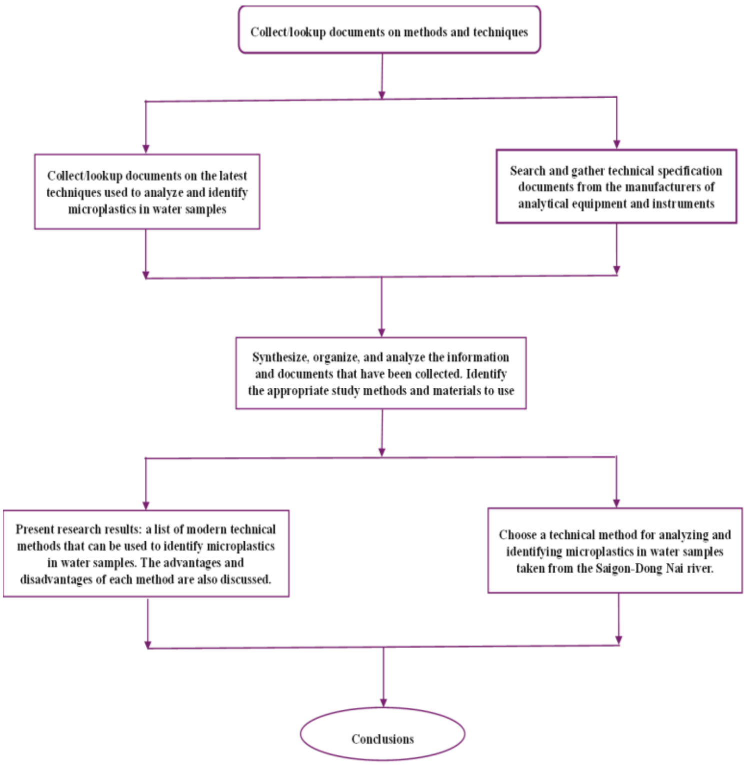

Figure 1. Flow chart outlining the process for implementing resear.

2.2. Study sites, sampling and surveys

The research was conducted at 18 locations in the Saigon - Dong Nai River basin in 2023. Of these, 13 locations are taken in the Saigon River branch from Dau Tieng lake to Ky Ha Rach junction (area near Soai Rap River) designated from S1 to S13 and 5 locations are in the Dong Nai River branch from Tri An lake to Dong Nai-Soai Rap River mouth with symbols from D1 to D5 (Figure 2).

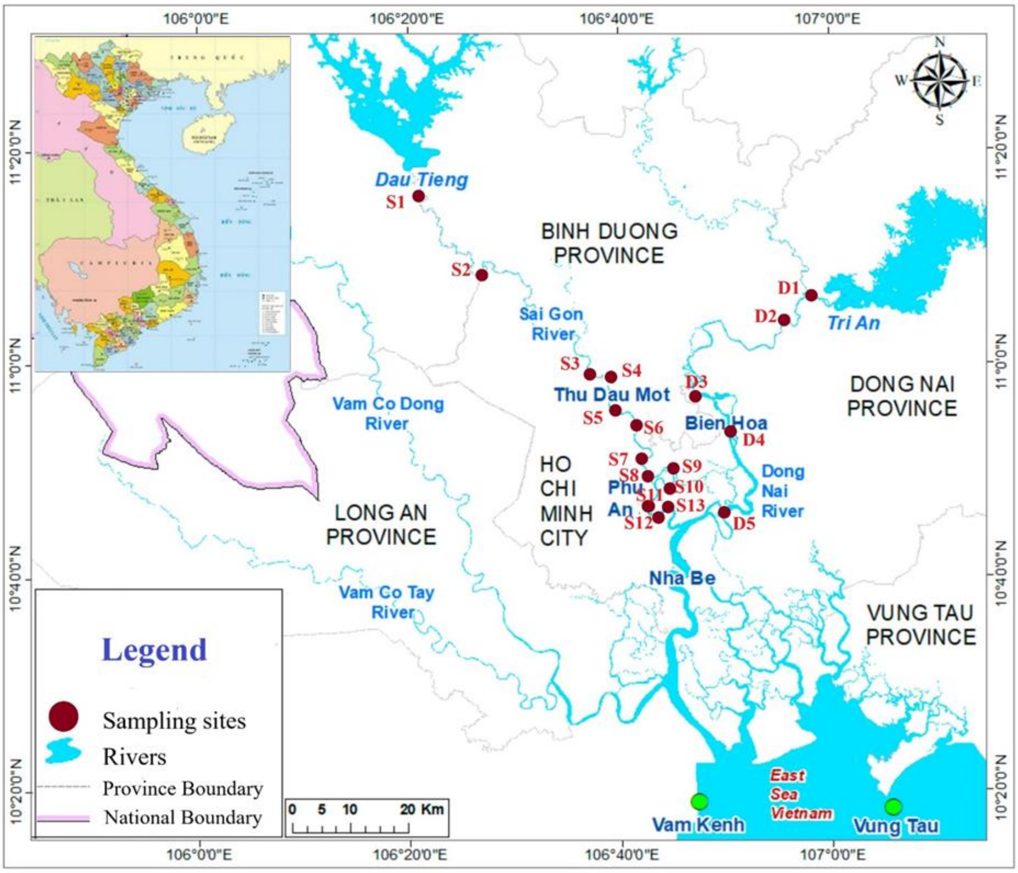

Figure 2. Sampling site.

2.3. Sampling method

Microplastic sampling in surface water was based on published methods of the National Oceanic and Atmospheric Administration [14]. The plankton net set (Neuston, 300 µm mesh) is attached to the Manta Trawl surface water microplastic sampling box (L×W×H = 30×30×15 cm) and anchored to the Neuston Katamaran floating buoy. The buoy is floated on the water surface to collect all solid objects floating on the surface water layer (from 0 to 15 cm) including plastic waste and other types of solids. A flow rate meter is also used to measure the water flow velocity at the time of sampling. Samples will be taken with a sampling time of approximately 30 minutes for each location. Once collected, the sample will be classified by hand to remove components > 5 mm in size. Then, these samples will be mixed together in a glass pot (washed and rinsed with 90% ethanol) to form a combined sample, and stored in a 2-liter glass jar with a tight lid. All water samples were transported to the nation lab and the southern institute of environment and circular economy (IECES) analysis room for analysis to identify microplastics.

2.4. Sample preparation methods before observation and identification techniques

To remove organic matter: 20 mL of 30 % H_2O_2 solution (hydrogen peroxide) and 0.05 M $FeSO_4$ (Fe II) solution were added to the device (beaker). The mixture was kept at room temperature for 5 minutes. Stir the mixture well and gently heat it on an electric stove (when you see air bubbles on the surface, take the cup off the stove and put it in a fume hood until the bubbles subside). Continue heating this mixture and adding 20 mL of 30% hydrogen peroxide until the reaction changes color from amber to light yellow.

To separate minerals and metals: Slowly add $ZnCl_2$ solution (d = 1.6 g/mL) into the sample mixture, stir well, then continue to drip $ZnCl_2$ solution into the tube to increase the density of the sample solution. This mixture is put into a centrifuge to completely separate microplastics from metals and minerals. Microplastics with low-density float to the surface of the $ZnCl_2$ solution (minerals and metals with a density greater than 1.4 g/mL sink to the bottom of the mixture). The detected polymer types can be used as the basis for similar inferences about their origin (Figure 3).

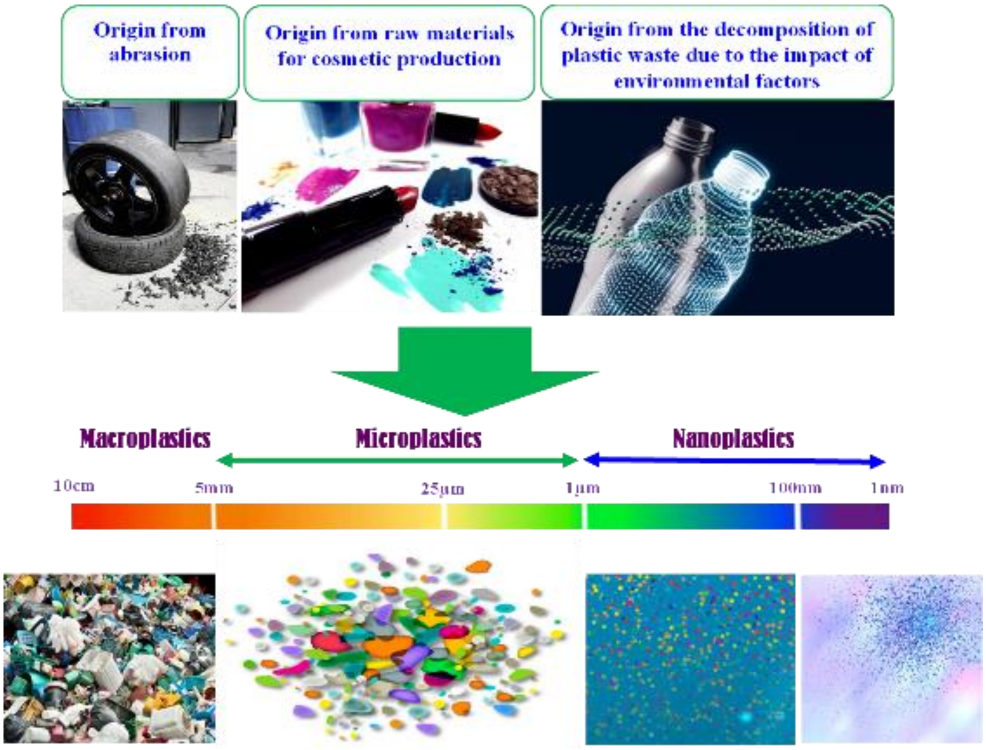

Figure 3. Size classification of microplastics and their relative origin.

2.4. Data analysis method

Calculation and statistical results are performed using Microsoft Excel Software and Spectrometer Technical Software.

3. Results and discussion

3.1. A review of analytical methods used in microplastics identification

3.1.1. Technique for identifying microplastics using electron microscope

Optical microscopy: Widely used to identify MPs in the > 100 µm range. Colorful microplastics can be easily identified using optical microscopy [15].

Electron Microscopy: Scanning Electron Microscopy (SEM), the sample was placed under a scanning electron microscope and all MPs present were counted and identified as flakes, pellets, fibers, films, or foams. SEM can provide very sharp, high-magnification images, even for very small particles, such as nanoplastics [16]; and transmission electron microscopes (TEM), devices that study solid-state microstructures, use magnetic lenses to produce magnified high-resolution images (up to millions of times), images may be produced on a fluorescent screen or on optical film, or recorded with a digital camera. Visual identification of microplastics via TEM may vary depending on the user. Additionally, the composition of additives such as Al, Ca, Mg, Na, and Si or antioxidants in microplastics is recorded [17]. Both have very high prices.

Gemstone microscope (polarizing microscope): it can be successfully used to identify polyethylene (PE). However, depending on the type of plastic, the structure of the plastic affects the transmission of polarized light when measuring. Therefore, this method is only suitable for measuring transparent microplastics [18].

3.1.2. Technique for identifying microplastics using thermal techniques

Differential scanning calorimetry (DSC): It is a thermal analysis technique that can confirm the physical properties of plastics. Because it is easy to melt MPs, it can only be used to identify some main types of microplastics such as PE and PP.

Thermogravimetric analysis (TGA): Equipment includes: a drying oven, micro-balance, temperature control system, and data acquisition. Confirmation of qualitative and quantitative information is performed by measuring the weight loss of the sample while heating at a specific rate under certain temperature conditions. But only polyethylene (PE) and polypropylene (PP) are clearly identified; polyvinyl chloride (PVC), polyethylene terephthalate (PET), and polyurethane (PU) are difficult to identify; polyamide (PA) and polyester (PES) were not identified.

Mass Spectrometry (Py-GC/MS - Pyrolysis Gas Chromatography/Mass Spectrometry): The chromatographic spectrum obtained from a sample is compared with the results of a known plastic standard to determine whether it must be plastic or not. This method can only determine PS well.

3.1.3. Microplastic identification technique using FTIR and Raman spectroscopy (Lazer)

Determining the type of microplastics in water samples was researched and analyzed using Fourier transform infrared spectroscopy (FTIR). When exposed to infrared radiation, microplastics absorb radiation at very specific wavelengths. The visual structure of the laboratory Fourier transform infrared spectroscopy (FTIR) analyzer used to analyze and determine the polymer type of microplastics in water samples is shown in Figure 4. The FTIR analysis technique for samples is a three-stage process: (i) The first stage is to record the FTIR spectrum of a new sample of MPs to obtain a basic FTIR trace; (ii) The second stage is to record the same FTIR spectrum of the used microplastic sample; (iii) The third and final stage is to subtract the new microplastic baseline, often called the new reference, from the used microplastic spectrum to obtain the difference spectrum.

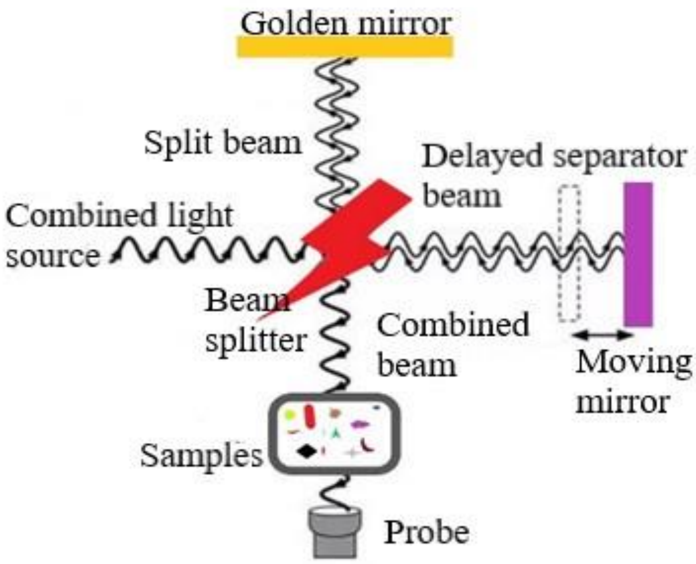

Figure 4. FTIR machine structure.

Both Raman and FTIR spectroscopy are capable of identifying microplastics. However, Raman spectroscopy has three distinct advantages when applied. The first is Raman spectroscopy which uses sub-micron wavelength lasers as the light source and, as such, is capable of resolving particles down to 1 µm or less. FTIR microscopy uses mid-infrared light as its source, resulting in a wavelength range that eliminates the ability to identify particles below 10 µm. The second is that, unlike IR systems, Raman microscopes are built around research-grade white light microscopes, making it easy to observe particles; The third is the ease of sampling. There is no need to choose between transmission, reflection, and the required technical ATR sampling, the Raman laser system focuses on the sample, and the spectrum is simply obtained by collecting the scattered light.

3.2. Identification of polymer types in surface water samples of Saigon - Dong Nai River using FTIR spectroscopy technique with ATR accessories

Data collection and analysis of microplastic polymers is also automated through research using FTIR-ATR. Figure 5 shows the Nicolet™ iN10 MX FTIR imaging spectroscopy microscope system combined with ATR accessories used to analyze and identify the polymer types of microplastics in this study.

Figure 5. Nicolet iN10 MX FTIR imaging microscope with ATR accessories: a) FTIR machine; b) The FTIR machine is connected to a computer system to record detected data; c) Signal processing and display software; d) Proceed to identify polymer types in the sample.

Applying modern FTIR technology Nicolet iN10 MX with ATR accessories to be able to more accurately determine microplastic components, more effectively for small-sized microplastics that cannot be determined by means of visual. The resulting spectra are compared with spectral libraries to find the closest match and determine the chemical composition. A concordance of 70% or higher was considered sufficient for confirmation. Polymer types of microplastics in the samples were confirmed through spectral peak data collected when running the samples (Figure 6).

In principle, reflection is the easiest technique because it does not require sample preparation or interaction between the microscope and the specimen. However, it can distort the spectrum, which can complicate the identification of plastic components. Therefore, before observing using polymer-type identification techniques, it is necessary to remove impurities such as minerals and perform Fentonization of the sample.

The two main characteristics that need to be studied for microplastics are physical properties (size, shape, and color) and chemical properties (polymer type). Any method that reliably measures both is suitable for analyzing microplastics in samples. Because it is difficult to obtain both types of characteristics using only one analysis tool, a combination of methods can be applied. The minimum limit size of microplastics, as in this article, is 1mm – 5 mm, this is an important factor to consider when choosing identification, and qualitative methods. The optical microscope is an essential tool for measuring physical properties.

Optical microscopes can only classify microplastics by color, making it difficult to identify the polymer type like a spectroscopic microscope. However, infrared spectroscopy is expensive and requires a technician to operate professional training. Thermal technology faces many limitations and is limited in the list of polymer types and destroyed microplastic samples (Figure 7).

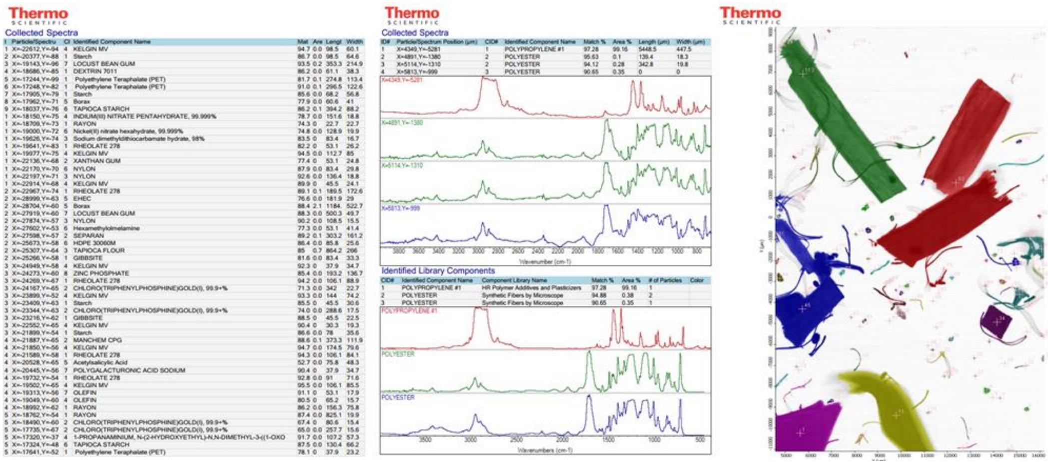

Figure 6. Interface of analysis results to determine the type of microplastics in water samples using the FTIR Transform Infrared Spectroscopy device on the screen.

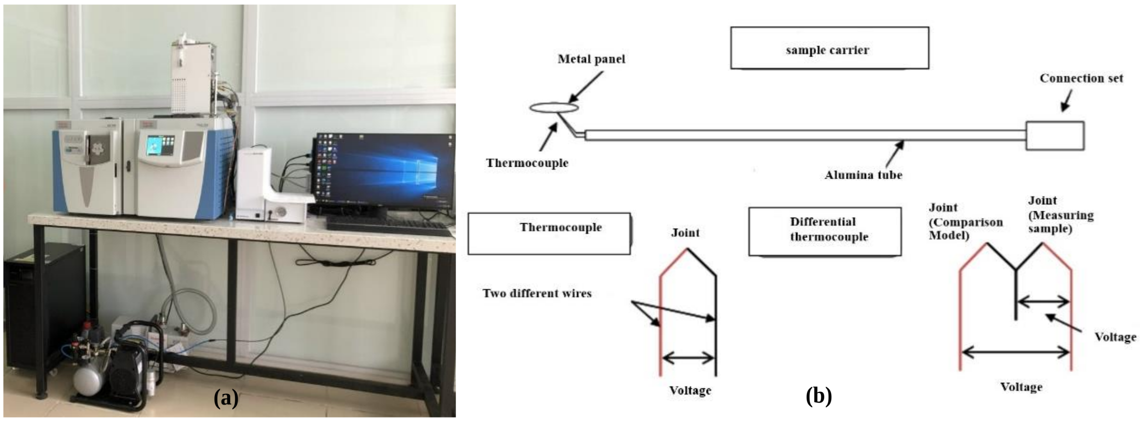

Figure 7. Thermal analysis techniques: a) Pyrolysi -GC-MS analysis identifies microplastics in the sample; b) Diagram of differential thermal analysis [19].

Pyrolysis -GC-MS analysis identifies isolated plastic particles from sediment samples such as PE, PP, PVC, PS, PA, PET, and chlorinated or chlorosulfonated PE. Thermal analysis provides an alternative to spectroscopy for the chemical determination of certain polymers. However, thermal analysis is a destructive method, which prevents further additional analysis of the sample. For large-sized microplastics that can be manipulated by hand (picking, counting...), magnifying microscopes and optical microscopes can be used because they will analyze the physical properties themselves along with a few additional tests (e.g. needle puncture) to determine the polymer type only relatively, lacking reliability. But if the size of the microplastic is < 1 mm and the minimum limit size is tens of micrometers, it is necessary to combine it with chemical analysis such as spectroscopy or thermal techniques to easily identify them according to the library of the system. Regarding convenience in processing, analysis time, and an abundant number of polymer types, choosing the µ-ATR-FTIR spectroscopy technique is the most optimal, especially for environmental samples.

If the minimum size is limited to a few micrometers, Raman (Lazer) spectroscopy should be a reliable technique for obtaining better spectra from particles < 20 µm in size. Although the µ-ATR-FTIR and Raman techniques are both very optimal, microplastics with too small sizes can still be missed or information lost in complex environmental samples with many impurities such as colorants, glues, catalysts, labels..., even for many unknown types of polymers such as weathering polymers. These methods are not recommended for routine monitoring studies today because they are very expensive, the equipment storage and preservation environment needs to ensure limited indexes of room temperature and humidity. Dust-proof, and above all, it requires a skilled technician to operate it. FTIR and Raman spectroscopy are powerful analytical tools for identifying microplastics in aquatic environments. There are many solutions, from simple point-and-shoot devices to complex imaging systems. The choice of system depends on the size of the particles being studied, on which the analysis to be performed is performed and the level of automation required. This information is summarized in Table 1.

Table 1. Information about infrared spectrum analyzers.

		FTIR + ATR	**FTIR + Smart Spot ATR**	**Point-and-Shoot FTIR**	**FTIR + Imaging + ATR**	**Raman microscope (Lazer)**
Microplastic size	5 mm	✓				
	1 mm	✓				
	500 µm	✓	✓			
	100 µm		✓	✓	✓	
	10 µm		✓	✓	✓	✓
	1 µm					✓
Just set the template manually		✓	✓	✓		
Automated analysis					✓	✓
Not affected by sample fluorescence		✓	✓	✓	✓	
Relative cost		Low	Medium	Medium	Hight	Very Hight

Data collection and analysis can be automated through the use of a microscope equipped with a motorized stage and associated software. Automatic analysis of this image using software can generate information about the identity, number, and size of each individual microplastic. The first result, though, is that images can contain large amounts of redundant data. There may be only a small percentage of the image data set that contains information about particles, the rest is filtered. The approach chosen is situational dependent on: i) How many particles are analyzed?; ii) In stock?; iii) In what area? Therefore, these advanced techniques prioritize identification and detection research and are limited in quantitative research, determining the density, color, and size of microplastics.

3.3. Polymer types detected in surface water samples of the Saigon - Dong Nai River using the FTIR-ATR method

As a result of FTIR transform infrared spectroscopy analysis, the study discovered more than 60 polymer types from microplastics found in surface water samples of the Saigon - Dong Nai river. This result is shown in Figure 8. Polymer types found in the samples according to chemical origin: VINYL-based plastic accounts for 3.29%; ETHYLENE-based plastic accounts for 40.8%; MUF synthetic glue-based resin accounts for 0.16%; TEFLON plastic (PTEF) accounts for 7.06%; PROPYLENE plastic accounts for 10.3%; OLEFIN synthetic resin (HIDROCACBON) accounts for 1.37%; AMINO synthetic resin accounts for 1.22%; STYRENE plastic accounts for 1.52%; NYLON accounts for 42.14%; The remaining 3.86% is other types of polymers.

The findings highlight how microplastics are already present in the water of these two tributaries from a range of plastic products such as packaging, nets, and single-use products such as straws and masks. The density of PE, PP, and PVC is very small, from 0.90 to 0.95 $g.cm^{-3}$ [20].

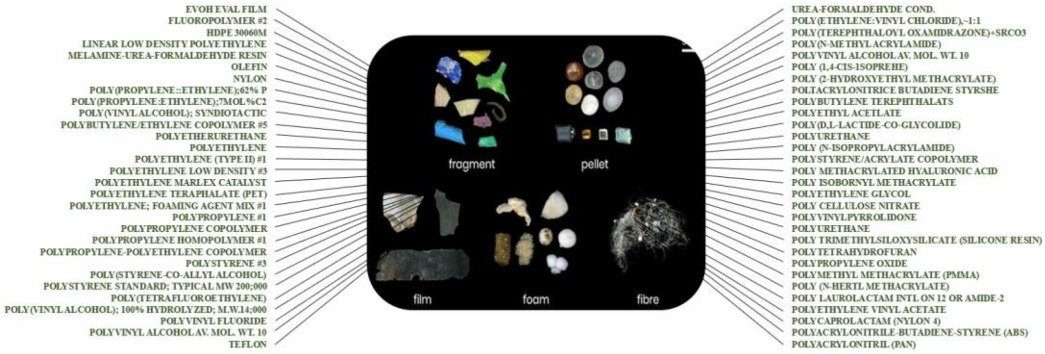

Figure 8. Polymer types detected in surface water samples of two branches of the Saigon and Dong Nai rivers.

They can float on the surface of river water, where the particles are then deposited on the sediment layer. Initial results of the study have identified microplastics present in the water environment. At the same time, the percentage of polymer-type radicals detected also represents a relative relationship from product to microplastic. Specifically, the NYLON plastic base belongs to the Polyamide industrial plastic group (PA plastic), with soft, smooth but waterproof properties, can withstand weather phenomena, and can resist natural influences such as mold or mildew insects, NYLON often produces artificial fiber products, doormats, tablecloths, raincoats, garbage bags, gloves, food wraps, and kitchen utensils,... these products have a high potential to decompose into fibrous and fragmented microplastics. In addition, PROPYLENE-based plastic is a hard, tough, and crystalline polymer thermoplastic produced from propene monomer (or propylene), often used to produce bottle caps, jars, and rice barrels, etc. that are difficult to decompose. If exposed to physical impacts and light environments that make them brittle and broken, they will mostly create microplastics in the form of hard pieces or granules. Similarly, ETHYLENE-based plastic is a flexible material that belongs to the polyolefin plastic group and plays an extremely important role in the plastic industry, producing many products for daily use such as food wrap, plastic straws, zip bags..., easily break and melt under sunlight and heat.

4. Conclusion

Different combinations of microplastic analysis methods will help identify microplastics in different complex environmental matrices. The need to identify microplastics from research projects are increasing, in many different environments such as water, air, and food to assess the risks and impacts of microplastics on natural ecosystems and human health, existing identification techniques need to be properly selected to reduce implementation time and effort. Although the optical microscope technique is simple, despite its low cost, it cannot identify microplastics. If the thermal technique is applied, it is limited because the polymer-type library is quite poor. After samples are analyzed using this technique, further experiments cannot be carried out because the microplastic sample is almost destroyed. Completely canceled and the price was also very expensive. By far the most popular spectroscopic technique for the analysis of plastics is total attenuation Fourier transform infrared spectroscopy (ATR-FTIR), a technique for the analysis of liquids. For samples smaller than 1mm, the ATR accessory can provide viewing and magnification capabilities, facilitating analysis of samples in the 1mm to 70 µm range. When the particle size drops below 100 µm, magnification is required.

There are two options, infrared microscopy and Raman microscopy (both are techniques also known as spectroscopic microscopy). For particles smaller than 10 µm, Raman is the preferred choice. Both FTIR and Raman are modern techniques but they are very expensive. In addition, the study has shown results on the presence of a variety of polymer types in the surface water of the two rivers Saigon and Dong Nai. FTIR imaging microscopy techniques combined with ATR accessories, specifically the Nicolet™ iN10 MX FTIR imaging spectroscopy microscope, more than 60 polymer types were detected. In particular, the highest is NYLON origin accounting for 42.14%, and ETHYLENE origin accounting for 40.8%. These are the two main sources in the production of packaging products, artificial fibers, raincoats, garbage bags, gloves, food wrap, kitchen utensils, and straws... these are flexible plastics, soft and smooth, easily disintegrate when exposed to rain, wind and heat from the environment, especially sunlight. Therefore, this issue should also be considered when research on microplastics is carried out. When performing identification, in order not to confuse polymer types with similar materials, it is also necessary to pay attention to those features to distinguish them. In addition, research should continue to identify and isolate microplastics from environmental samples through the introduction development and updating of new polymer types into the library spectrum, which is a major limitation because it requires coordination. Coordinate with manufacturers and update software systems.

Author contributions: Developing research ideas: H.P., H.T.N.H.; Choosing research methods: H.P., H.T.N.H., T.N.N.; Data processing: H.T.N.H., T.T.M.H.; Sampling: H.T.N.H., T.N.N.; Writing draft of article: H.T.N.H., T.N.N.; Editing the article: H.P., H.T.N.H.

Acknowledgments: This research was conducted under the sponsorship of the research project Institute for Environment and Circular Economy in The South (IECES).

Declaration: The authors warrant that this article is their own work, has not been published elsewhere, and has not been copied from previous research; There are no conflicts of interest within the author group.

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