DETECTION AND QUANTIFICATION OF MICROPLASTICS

A M.Sc. Dissertation report by:

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APRIL 2022

DETECTION AND QUANTIFICATION OF MICROPLASTICS

A DESSERTATION REPORT

Submitted in Partial Fulfilment

Of

The Degree of M.Sc. (Analytical Chemistry)

By

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To the

School of Chemical Sciences

Goa University

Goa 403206

April 2022

CERTIFICATE

This is to certify that **Harshad Naik** of M.Sc. (Analytical Chemistry) has successfully completed the dissertation work on the topic **Detection and Quantification of Microplastics** in the year 2021-2022. It is further certified that this project is the individual work of the candidate.

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Abstract

Microplastics are widely spread contaminants, present in all environmental parts. However, knowledge on sources, fate and environmental concentration over time and space still is limited due to the difficult and varied analytical procedures currently used. As an essential source of aquatic products, aquaculture industries are certainly subjected to the pollution of microplastics. MPs can be quantified and detected mainly by visual inspection, spectroscopy, or thermal analysis. However, this review focuses on the detection and quantification of microplastics using different analytical techniques such as Raman spectroscopy, FTIR spectroscopy and so on. From Nile red straining the shape and size of the MPs can be revealed by studying their CLSM images. Since this method is easy it can be greatly applied. From impedance spectroscopy we are able to detect and count the number of microparticles in solutions. By using Raman spectroscopy technique it was easy to determine the structure and composition of the microplastics. This technique has high reliability, high throughput analysis and requires less amount of sample which makes it advantageous over other techniques. Q-NMR technique can also be used to quantify microplastics by measuring their signal area. Another spectroscopy method that can be used for microplastic quantification is Fourier transform Infrared spectroscopy in which the bond of the structure can be compared with the standard sample and can be identify accordingly. This method has simple operation and accurate identification. To quantify microplastics with more accuracy and ease the combination of different methods can be used such as pyrolysis GC/MS. In this method chemical composition of polymers can be found out by studying the pyrograms developed by it. Different thermal analysis techniques can also be used for detection microplastics which includes thermogravimetric analysis in which certain plastics can be identified by thermoanalysis.

Introduction

Microplastics are small plastic pieces <5mm intentionally produced to be used in consumer products and in activities as abrasives or resulting from the breakup of larger objects highly persistent contaminants potentially harmful to organisms or ecosystems.[1] MPs can migrate in various environmental parts, such as air, soil, oceans and freshwater. They are easily ingested by organisms and translocated to higher trophic level through food web.

MPs are classified into primary MPs, and secondary MPs. The 1° MPs are derived from microbeads in cosmetics, cleaning products and air-blasting media which can directly enter the environment, while the 2° MPs are derived from the decomposition of layer plastic pieces.[2]

Microplastics are usually visually identified with a microscope. However, the method has low reliability, especially for small, transparent and fibre-type particles. SEM-EDS can not only provide high-resolution images of the surface texture of microplastics, but also examine the elemental composition of the particles, particularly identifying carbon-dominant plastics from inorganic particles.[3]

In this review, we utilize CLSM images stained with Nile red to tell their shape and size, focal plane array-based reflectance Micro-FTIR imaging to identify different types of microplastics, flow through quantification of microplastics using impedance spectroscopy and so on.

Sampling and collection of microplastics

Collection of microplastics is the first step of sampling methods of MPs. The most common sampling methods of MPs include selective sampling, volume-reduced sampling, and bulk sampling. MPs are directly extracted from samples by visual identification, which is defined as selective sampling. Volume-reduced sampling indicates that samples are filtered and sieved, and thus the target components can be used for additional analysis at the sampling location. Bulk sampling does not separate components on-site and keeps all samples. For large water body areas, static sampling is carried out at each sampling point, and filtering sample collection is generally selected.

Microplastics in aquaculture environment can be collected from water surface or the water column at certain depths. For surface water sampling, neuston nets, and manta trawls are most commonly used tools, but for water column sampling, near-bottom trawls, multiple opening-closing nets, continuous plankton recorders, bongo nets, and plankton nets are the main tools.

Some alternative equipment are occasionally applied in surface water or water column sampling for MPs, such as water intake pumps, water collection bottles, or plankton traps. The smaller the size of the mesh, the smaller the particle size of the MPs.

In the aquatic environment, the depth of the water should be considered for sampling, and it is generally acknowledged that the abundance of MP surface is higher than that from water depth of 1-2 m.

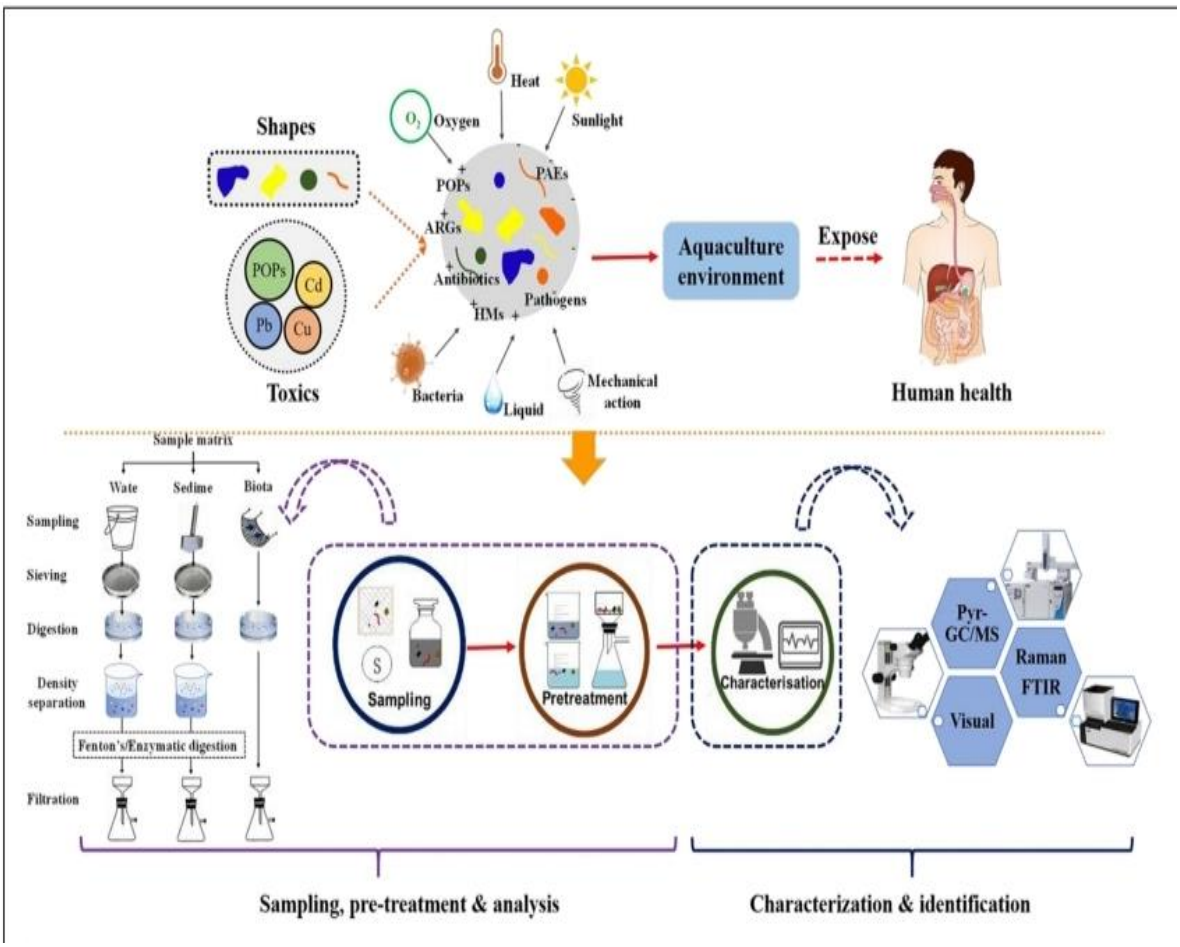

***Figure 1*: analysis process of microplastics**

METHODS

1. Detection of microplastics using Nile Red Straining

Plastics consist of hydrocarbons derived from petroleum, natural gas or biomass and are hydrophobic. Lipophilic dyes can be used to visualize microplastics under a fluorescence microscope. Nile Red (9-diethlyamino-5H-benzo [α] phenoxazine-5-one), a fluorescent dye, has been used to strain neutral lipids in biological samples. NR is a hydrophobic fluorophore that specially binds to neutral lipids and is strongly fluorescent only in the presence of a hydrophobic environment.[4] From this applicability it is found that microplastics appear irregular shape and the fragments of the microplastics are in micron scale. This method is useful for detection of polyethylene, polystyrene, polyvinyl chloride and polyethylene terephthalate plastic particles.[5]

In case of staining MPs in solution firstly the staining solution is prepared which includes organic solvent that dissolves NR. The solvents used for this method are methanol, chloroform, acetone, and n-hexane. Next is suspending MPs in the staining solution (fig.2b). The intensity of the fluorescence signal can be enhanced by heating and cooling which also inhibit leaching of the dye.[6] The third step is filtering the staining solution to get the fluorescence-stained MPs and washing them with deionized water to get rid of the adsorbed dye (fig.2c). Then the fluorescence stained MPs are obtained and they usually show red or purple colour when NR is used as a dye (fig.2d).

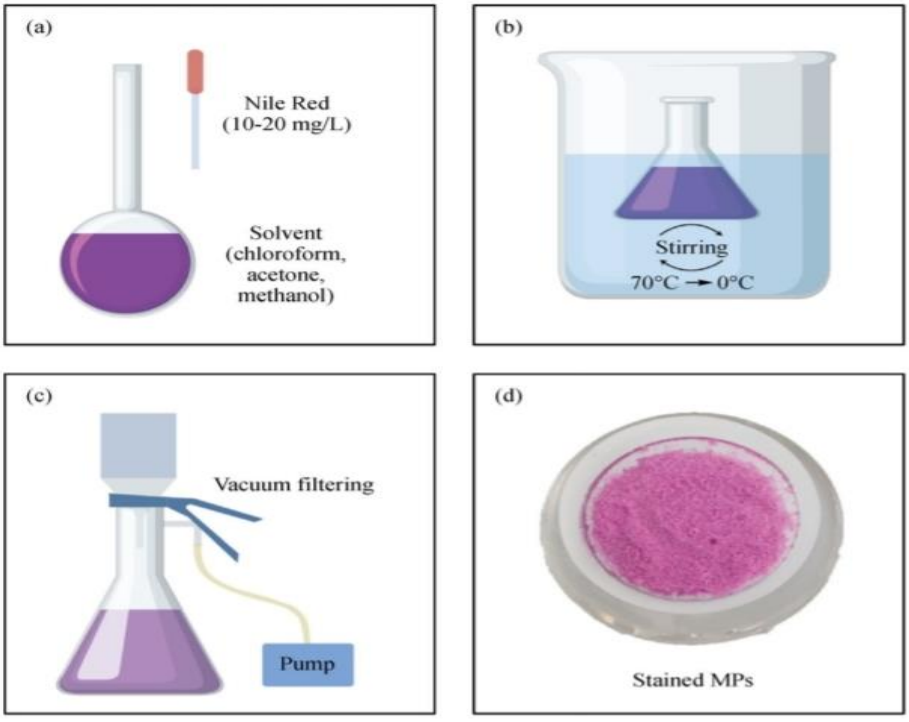

Figure 2: **MP staining process in solution; a) adding Nile red to solvent, b) staining in solution while heating and cooling, c) vacuum filtering, d) Nile red stained MPs**

The dye absorbs onto the plastic surfaces and give them fluorescent when irradiated with blue light. Fluorescence emission is detected using simple photography through an orange filter. Image analysis allows fluorescent particles to be identified and counted.[7]

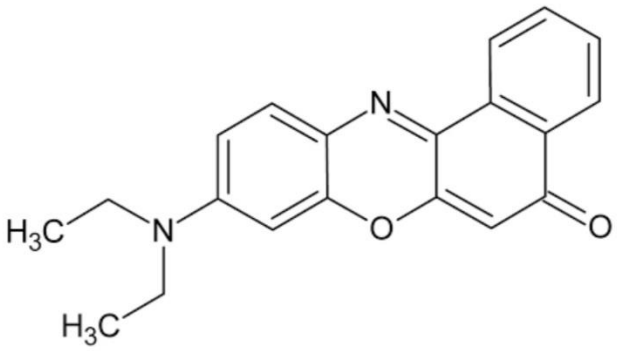

Figure 3: **Nile Red**

Limitations of NR straining

The main limitations in applying the NR straining method to samples is the co-straining of natural organic material. It is therefore important to remove natural lipids and organic matter from the samples before NR straining.

Also the number, shape and type of NR-strained organic fragments can vary from sample to sample.

2. Quantification of microplastics using impedance spectroscopy

For high-throughput flow-through measurement of microplastics impedance spectroscopy is used due to its low cross-sensitivity to biological particles. The overall aim is to apply this technique for detecting and counting the number of microplastics present in an aqueous solution.[8]

The secondary goal is to determine its use for sizing the microplastics as they are detected. In flow cytometry applications, impedance spectroscopy is used to understand the electrical properties of single cells at high throughput, with recent results differentiating red blood cells and plastic beads at 200 cells/s. Electrodes monitor the impedance change as particles pass in a flowing medium, such as phosphate-buffered saline, tap water or salt water.

By using Ohm's law, the impedance relates a voltage source to the magnitude and phase of current passing through a circuit element as a function of frequency. At low frequency the impedance change is proportional to a particles volume and is used in coulter counters for sizing. At higher frequencies, particle internal properties may be measured, such as membrane capacitance or

cytoplasm conductivity. Impedance spectroscopy has been applied to blood analysis, tumour cell identification bacteria detection and plankton discrimination. Plastic beads which are used for testing and size calibration, are differentiated from biological particles through a combination of high and low frequency measurements.

In impedance flow cytometry, particles are typically 1-25µm. For microplastic analysis, it is necessary to expand impedance spectroscopy to cover a larger size range (1-1000µm).

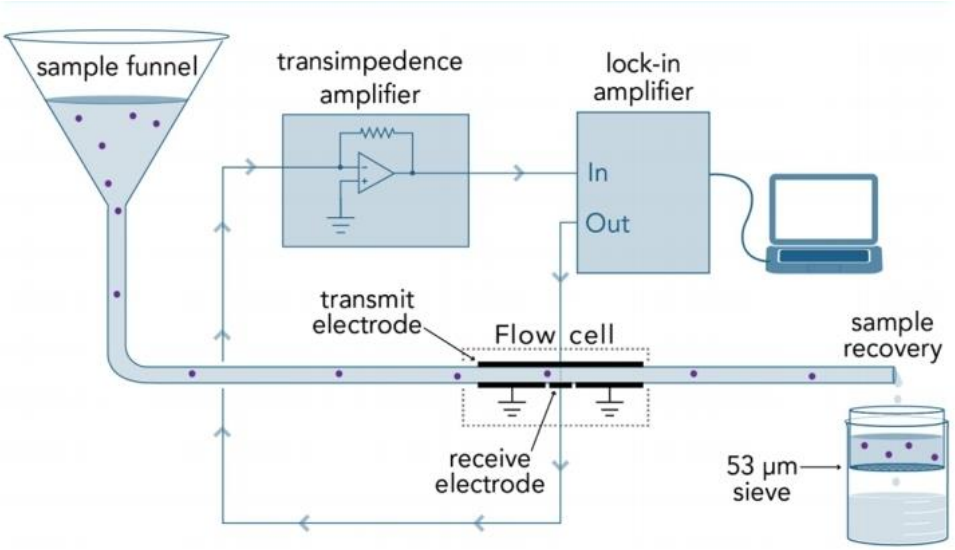

Figure4: **Impedance spectroscopy experimental setup**

In this experiment particle-spiked samples are added to the sample funnel. Clean water is then added periodically to maintain the water level until all particles are flushed through. The flow is gravity fed. A transimpedance and a lock-in amplifier are used to monitor the impedance between the transmit and receive electrodes at six frequencies simultaneously. The changes in impedance due to particles passing through the flow cell are detected in post-processing. After passing through the flow cell, the particle-spiked water is filtered through 53µm sieve and microplastics are visually counted.

3. Detection of microplastics using Raman spectroscopy

Raman spectroscopy is a vibrational spectroscopy technique based on the inelastic scattering of light. Based on the scattering spectra of different frequencies of the incident, it is possible to obtain the molecular structure of substance. Characteristic spectral fingerprints can be achieved by detecting the molecular vibration of sample through Raman spectroscopy and the composition of the sample is identified by comparing it with a known reference spectrum. Raman spectroscopy also

allows the observation of local microscopic features. Raman spectroscopy with a high spatial resolution (<1µm) is not sensitive to interference signals from water and atmospheric carbon dioxide. The position of stokes line is used for qualitative analysis and the intensity of the line is interpreted to determine the concentration of the sample.[9] The main advantages of this technique are the high reliability, the possibility for high throughput analysis, and low sample amount requirements. A combination of Raman spectroscopy with microscopy can be useful to identify MPs down to 1 µm in size.[10] MP particle size is determined by fitting an ellipse geometry on the particle contour resulting in a long and short particle dimension.[11]

The fluorescence of the sample influences the Raman spectroscopy signal. Raman spectroscopy has the advantages of higher spatial resolution and no interference from water, and some information can be obtained using Raman spectroscopy rather than infrared spectroscopy.

Limitations of Raman spectroscopy

The fluorescence effect from the pigment, additive, or containment in the environmental sample affects the measurements of sample with Raman spectroscopy, and the detection time of Raman imaging is remarkably higher than FTIR. Also the fluorescent samples excited by the laser cannot be measured as they prevent generation of interpretable Raman spectra.[7]

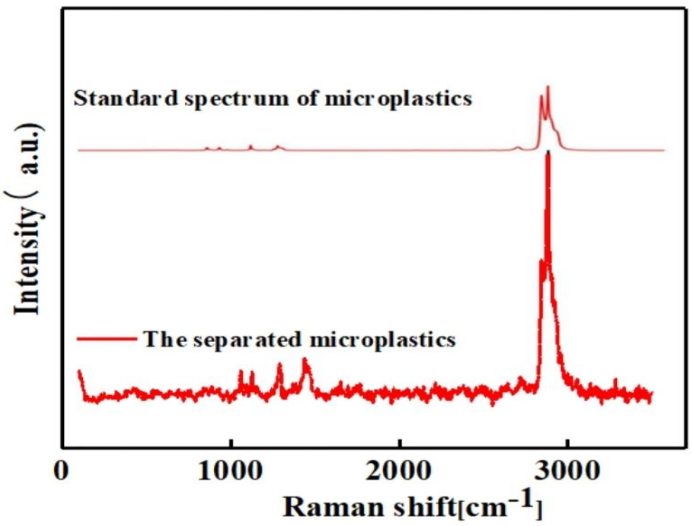

Figure 5: **Raman spectra of the standard spectrum of microplastics and the separated microplastics**

4. Quantification of Microplastic particles by Q-NMR

Quantitative determination by Q- NMR is based on the proportional relationship of integrated signal area and number of resonant nuclei. Q-NMR can be described as a precise quantification method since with this method a high quantitative accuracy of >98% can be achieved.

For analysis of MP particles in environmental samples, the calibration curve method is best suited because the exact composition of analytes does not need to be known which the case for most MP samples is.

Limitations of Q-NMR

The disadvantage of quantitative ^{1}H NMR spectroscopy is the dissolution of the analysis in a suitable deuterated solvent leading to a loss of size information of the MPs. Therefore it is important to find suitable conditions for the analysis of MP particle by means of Q-NMR in order to dissolve different types of polymers (PE, PP, PET, PS, PVC…).[12]

5. Quantification of microplastics by pyrolysis-GC/MS

Pyrolysis-gas chromatography in combination with mass spectrometry can be used to evaluate the chemical composition of potential microplastic particles by analysing their thermal degradation on products.[13] The pyrolysis of plastic polymers results in characteristics pyrograms which facilitate an identification of the polymer type. The polymer origin of particles is then identified by comparing their characteristic combustion products with reference pyrograms of known virgin-polymer samples. If a thermal desorption step precedes the final pyrolysis organic plastic additives can be analysed simultaneously during pyrolysis- GC/MS runs.

Pyrolysis GCMS can also be applied for quantitative trace analysis of MP on a polymer specific level if the pyrolysis conditions are highly reproducible to generate a consistent composition of pyrolysis products.[14]Compared to solvent extraction techniques, Py-GC/MS has the advantage of being able to analyse the polymer type in one run without using any solvents and without background contamination.[15]This method has an appropriate degree of sensitivity for analysis of plasticisers, antioxidants and flavouring agents in microplastic particles with sample size below 350 µg.

Compared with conventional GC/MS, pyrolysis-GC/MS has an additional micro-furnace pyrolyzer mounted vertically on the GC instrument, which allows for pyrolysis of a small quantity of samples as

shown in figure below. The operating temperature for the pyrolyzer is typically between 500°C and 800°C. The gaseous effluents that evolve from the micro-furnace pyrolyzer flow directly into the GC column, and the separated effluents through the GC column are identified with the ionization of each molecule using the MS detector.

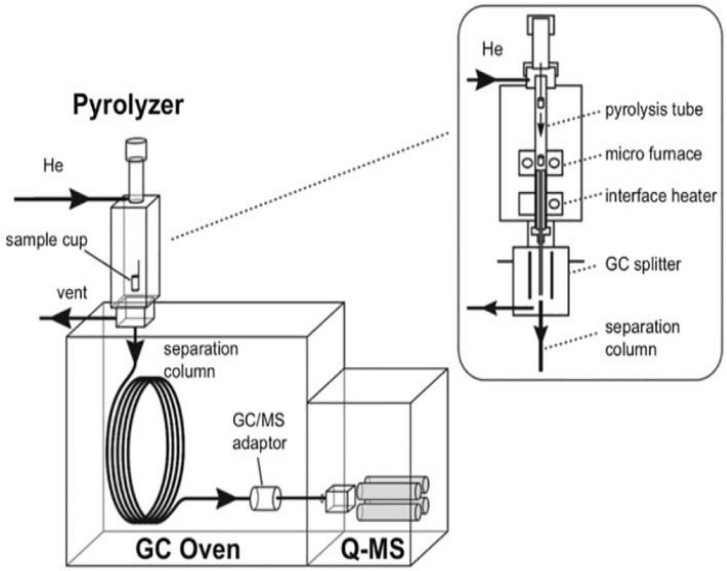

Figure 6: **Pyrolysis GC/MS System**[16]

Limitations of pyrolysis-GC/MS

Although the pyrolysis- GC/MS system allows for a relatively good job of potential microplastics to polymer type it has the disadvantage that particles have to be normally placed into the pyrolysis tube. Since the particles of a certain minimum size can be manipulated manually this results in a lower size limitation of particles that can be analysed. Furthermore, the technique allows only for the analysis of one particle per run and is thus not suitable for processing large sample quantities, which are collected during sampling or routine monitoring programs.

It does not allow to determine the number, type or morphology of microplastics, as it only provides the mass of polymer per sample, thus requiring pre-selection of microplastics by optical techniques.[17]

6. Quantification of microplastics using Fourier transform infrared

FTIR mainly provides chemical bond information of compounds. The generation of peak types and specific spectrum rely on the bond structure. In comparison with the standard library, MPs can be distinguished from other organic and inorganic substances. The composition of MPs can be identified if the matching degree of MPs detected by infrared spectroscopy reaches more than 70% with the standard library.

For this technique it is necessary to remove the (in) organic matrix using one of the many different treatments available. Afterwards, the sample is concentrated onto membrane filters.[18] FTIR used to MPs analysis mainly has three modes: transmission, reflectance, and attenuated total reflection. In transmittance mode, the investigated particles need to be sufficiently thin (<100 μm) to avoid total absorption in the FTIR spectrum.[19] There are two types of FTIR for MP analysis, namely mercury cadmium telluride (MCT) single mode and microscopic equipped FTIR. The MCT single mode can only analyse single and bulk samples by detecting the sample with an attenuated total reflectance tip, while microscopy equipped FTIR can identify the samples with heterogeneous components of MPs. The microscopy equipped FTIR analyse samples with two different modes: ATR and focal plane array (FPA) detector mode. In ATR mode, individual particles are identified by microscopy and subsequently detected using an ATR tip.

The FTIR method was also widely used for the characterization of MPs because of simple operation and accurate identification. Micro-FTIR can be used to identify MPs isolated from fish tissues and the main polymers that can be found such as PET, PES and PE. Micro-FTIR not only improves spatial resolution but also enable the detection of smaller plastic particles.

MPs in the environment can also be identified using Raman spectroscopy and FTIR, and can be capable of being detected in different spectral ranges for synthetic polymers.

Limitations of Fourier transform infrared spectroscopy

This method is time-consuming and it is easily affected by plastic inhomogeneity and material aging. In addition, some particles with size <20μm cannot be detected.

7. Detection of microplastics using scanning electron microscopy

In this method an intensity electron beam is generated and scans the sample surface. High resolution images (< 0.5 nm resolution) of the surface details are produced because of the interaction between the electron beam and the sample. Microplastics can be identified from the sample particles by comparing their surface features.[20] The mechanical degradation patterns of microplastics can be obtained by examining the surface texture such as grooves, pits, fractures, and flakes on the microplastics using SEM.

The combination of SEM and energy-dispersive X-ray spectroscopy (SEM-EDS) can provide information on the elemental composition of particles.

Limitations of Scanning Electron Microscopy

It is a time-consuming method in sample preparation and observation. Therefore, SEM is not suitable for the identification of a large number of microplastics.

8. Quantification of microplastics using Focal Plane Array-Based Reflectance Micro-FT-IR Imaging

In this method a pretreatment of sample is done using 30% hydrogen peroxide to remove biogenic materials and to facilitate filtration, and is done to successfully image and identify different microplastics types. The FPA based micro-FT-IR spectroscopy also provides a considerable reduction in analysis time since samples that could take several days to be mapped using a single-element detector can be imaged in less than 9hr.[21]

9. Detection of microplastics using hyperspectral imaging

Every physical object emit radiation in the electromagnetic spectrum on different wavelength due to their chemical composition. Spectral imaging is a combination of both spectroscopy and imaging. HSI imaging refers to the gaining of an image of the target sample. Multiple particle can be identified simultaneously by HSI. Depending on the interaction of different wavelengths of light with chemical compounds in a sample a distinct representative spectrum of the sample is generated. When combined with machine learning algorithms, HSI may produce faster results than other MP identification technique.

Polymer composition is necessary for MPs identification by HSI. In order to analyse spectral images of MPs more effectively using PCA (algorithm), noise signal from the background is usually removed by limiting the spectral window to areas where MP show less degradation effects on spectral images. This spectral window contains the overtone which have a high signal strength and hence optimal for an initial analysis of the samples that are prone to exhibit higher degradation effects as compared to their reference.

Limitation of HSI

Large amount of data generated that often contains redundant data, not helpful towards the analysis thus requiring complex data analysis methods to be employed.

10. Quantification of microplastics by thermogravimetric analysis

Thermogravimetric analysis monitors the thermal stability and portion of volatile compounds in a sample. It measures the mass change of samples as a function of thermo analysis temperature to quantify the volatile compounds, and the derivative of the mass-change curve determines the mass-loss rate.[22]

Different plastic materials exhibit specific thermoanalytical behaviours in a broad temperature range, allowing for the identification of specific types of plastics when waste materials are fully separated and purified from the environmental matrix.

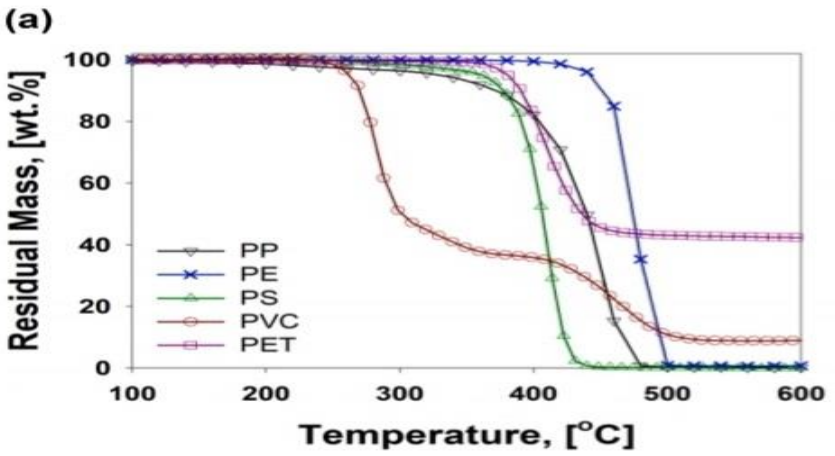

Figure7: **Thermogravimetric analysis of different plastics**

For quantification of microplastics thermogravimetric analysis can be coupled with differential scanning calorimetry. The limitation of this method is that it can only identify polyethylene (PE) and polypropylene (PP) due to destructive nature of the methods and number, size and form of the particles remain unknown, which is the drawbacks.[23]

11. Detection of microplastics using Resonance Microwave Spectroscopy

Microwave spectroscopic characterization of microplastics contained samples is based on the permittivity contrast between the blank and the microplastic contaminant. Microwave spectroscopy consists of low cost sensors, their small size, rugged design for real-time, in-situ operation makes them advantageous.[24] This features are attractive for the in-situ microplastic detection and quantification in the flexible sea monitoring platforms, sewage and wastewater processing plants, smart households sensing for detecting microplastics in drinking water and food.

A microwave reflectometry sensing system consists of a signal source (TX), microwave probe or antenna electromagnetically (EM) interacting with a sample under test, and a receiver (RX) which combines the reflected signal from the probe and a reference signal from the source and generates an interferometric output signal. The reflected signal from the probe is characterized by the S_{11} parameter and carries the information about the sample-under-test permittivity. Microwave reflection spectra of the sample are obtained by sweeping the frequency of the source TX and collecting the S_{11} data, from which the material parameters (such as microplastic concentration in the sample) can be extracted using a suitable mathematical model. The two important parameters of the microwave reflection spectrum are the resonance frequency where the magnitude of S_{11} is minimal and Q-factor defined as the ration of the resonance frequency to the resonance bandwidth at 3dB level.

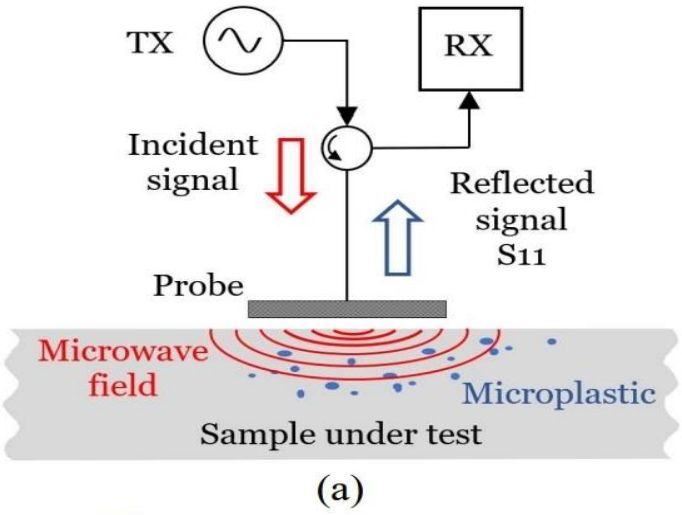

Figure8: **Microwave Reflectometry diagram**

12. Detection of microplastics using differential scanning calorimetry

It measures the amount of the energy required to increase the temperature of a sample and a blank reference as a function of temperature. When a phase change occurs between a solid and a liquid, additional latent heat is required in the sample compared with the blank reference. The different heat requirements of the sample and reference holders during phase change provide information on the glass transition region, crystallization and melting temperatures of polymeric materials.

From figure 6 it can be seen that PE and PP has relatively sharp and distinctive melting temperatures near 101°C and 164°C, respectively. But other plastics have broad endothermic peak ranges around their melting temperatures. The overlapping melting temperature ranges for PET, PA, PES, PVC and PU only allows for the identification of PE and PP in plastic mixture samples (from figure 7).

The thermoanalytical behaviours of plastic particles are affected by thermolysis conditions and the status of plastic particles such as additives, particle size, degree of polymerization and crystallinity.

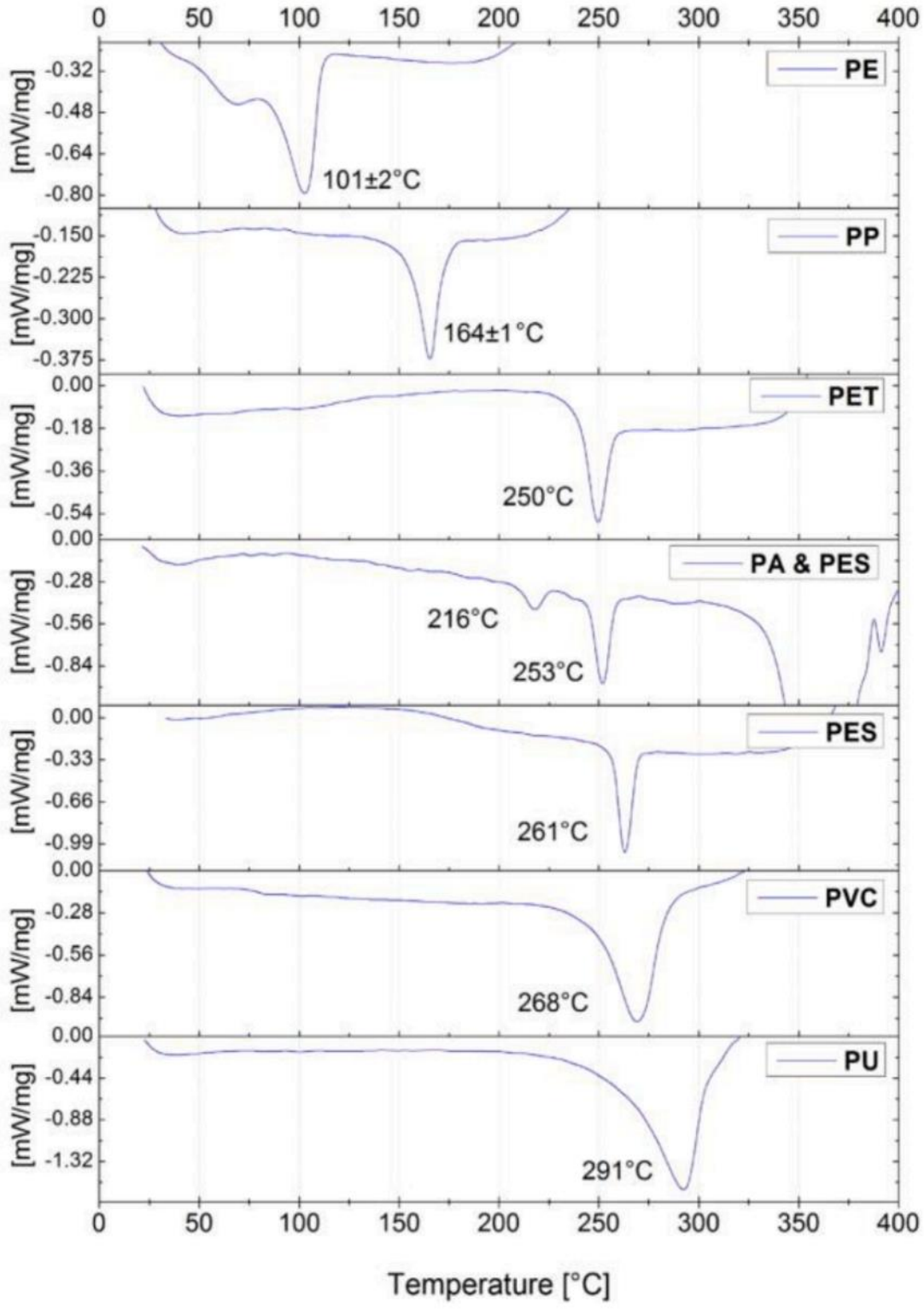

Figure6: **DSC of different plastics showing endothermic phase transition heat flows and melting temperatures**

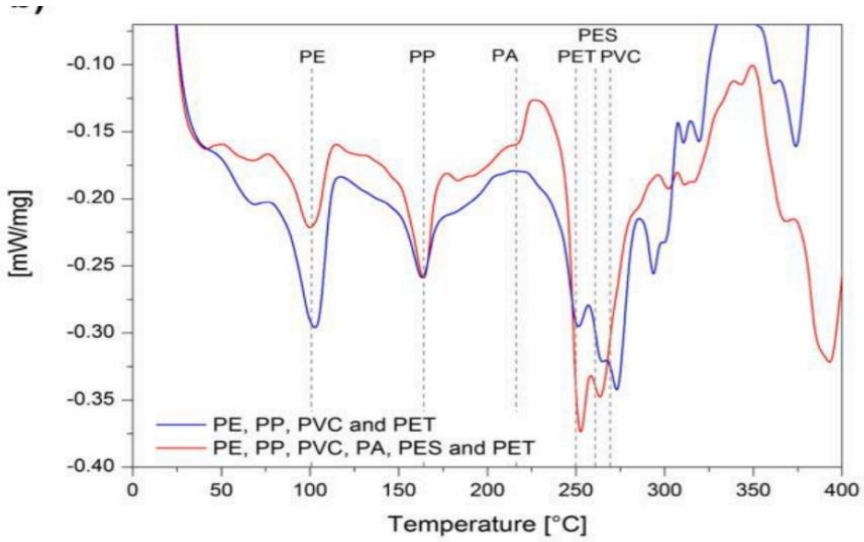

Figure7: **DSC signals of polymer mixtures**

13.Quantification of microplastics using accelerated solvent extraction

Accelerated solvent extraction is a method that is used to recover semi-solvent organic compounds from solid materials. Microplastics may be automatically extracted using this method.

ASC is characterised by automation, low cost and high efficiency. It has advantage of quantifying total mass content of microplastics regardless of size, shape and colour.[25]

Conclusion

This work has shown the different methods and methodologies for detection and quantification of microplastics that exist in the environment which are often responsible for causing harmful effect for the same. Despite having some limitations with the methods the results provide the feasibility and applicability needed for its purpose which includes sampling, shapes, sizes, abundance, surface morphology and surface elemental characteristics. The combination of multiple analytical techniques provided an accurate approach for microplastics identification and characterization.

Acknowledgement

I would like to express my special thanks of gratitude to my guide Dr. Rupesh Patre for their guidance and support in completing my project.

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