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Review

Identification and characterization of micro-plastics in the marine environment: A mini review

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ABSTRACT

Micro-plastics (MPs) are an environmental threat that has been gaining importance lately with an increasing number of studies demonstrating that they are a larger threat than previously thought. Scientists around the world have used a wide number of methods in their studies and they have adapted changes in response to the specific nature of the research undertaken. This article provides an account of the historical development of the MP menace, development of methods and tools used in MP research and also describes the challenges that are faced to further advancement to take place. The article is categorized into various sections that include history, sources, isolation, extraction, and characterization of MPs. Among the thermal characterization techniques, direct pyrolysis mass spectrometry and secondary ion mass spectrometry, which are widely used to characterize the plastics, but not utilised so far in this field are also highlighted for future direction.

1. Introduction

1.1. Plastics: a complex history of market demand and dominance

In essence, plastics are a long chain class of Organic polymers that have a high Molecular Weight. The organic mass that the common plastics compose of are largely derived from the fossil fuel feed (Resins, 2015). In the process of deriving such a plastic that would suit a given application, plastic resins are added with various substances to render them with properties such as increased strength, greater durability, light weight and insulation (Thermal and electric). The substances added may well include fillers, plasticizing agents, stabilizers (UV and Thermal), antimicrobial agents, coloring dyes, etc., and the product may take up forms such as foams, sticking substances (Adhesives), fibers, films and other moulds in solid.

The introduction of such a developed polymer occurred as early as the mid-nineteenth century, where accelerated commercial production followed towards the finish of the second-world war. The development of several variants of plastics happened during the early twentieth century, in line with the exponential growth that followed the 1950s. One estimate for the prevalence of plastic forms today is that about seven types of commodity thermoplastics account for roughly 85% of the total plastic available in the markets globally (Resins, 2015). Another estimate puts the total plastic produced in terms of weight as of 2014 at 3.11×10^8 metric tonnes (Europe, Plastic, 2015).

The plastic material provided a range of solution to the market problem of packaging material. In the US alone, packaging plastic accounts for a little over one third of the market demand, and in a similar trend, such plastics that serve very short-term needs account for the larger chunk of market demand. Another estimate states that a low 8.8% of the total consumer plastic is recycled (EPA, US, 2014), this regardless of the greater fraction these plastics hold in the total waste generated (Estimated at 12.8% of solid waste mass collected at the municipality level, 4) and, the very convenient and feasible process of recycling that consists of a breaking down stage followed by re-melting (Andrady, 2015). A notable finding was that Europe, known for its greater recycling capabilities of plastic, only has a 30% recycle rate (Europe, Plastic, 2015), in spite of being an advanced and self-sufficient region. This can be accounted in for by the nature of use of consumer plastics that pose a challenge in its recycle abilities such as processing damages, improper discarding, In addition there are possibilities of feed contamination, and also the marketing difficulties of recycled plastics (Andrady, 2015). Publication trend in Microplastics (MPs) from 2011 to 2020 searched in sci-finder (Fig. 1). This indicates that this field is booming in an unprecedented manner.

The debris found in the marine environment is composed of those that were transported or readily dumped in the ocean such as solids that were manufactured for a relevant purpose. They may take up several

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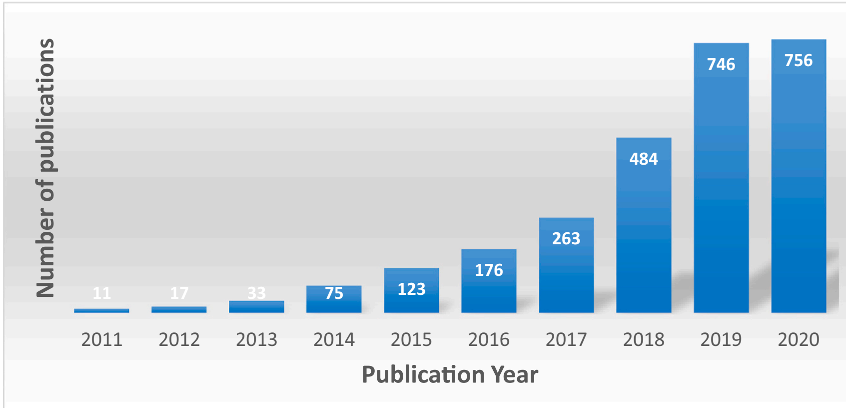

Fig. 1. Publication trend in MPs from 2011 to 2020. The data is searched in Scifinder, search hint MPs as keyword and marine as refine word. Only published in English are included here. Assessed on 28th July 2020.

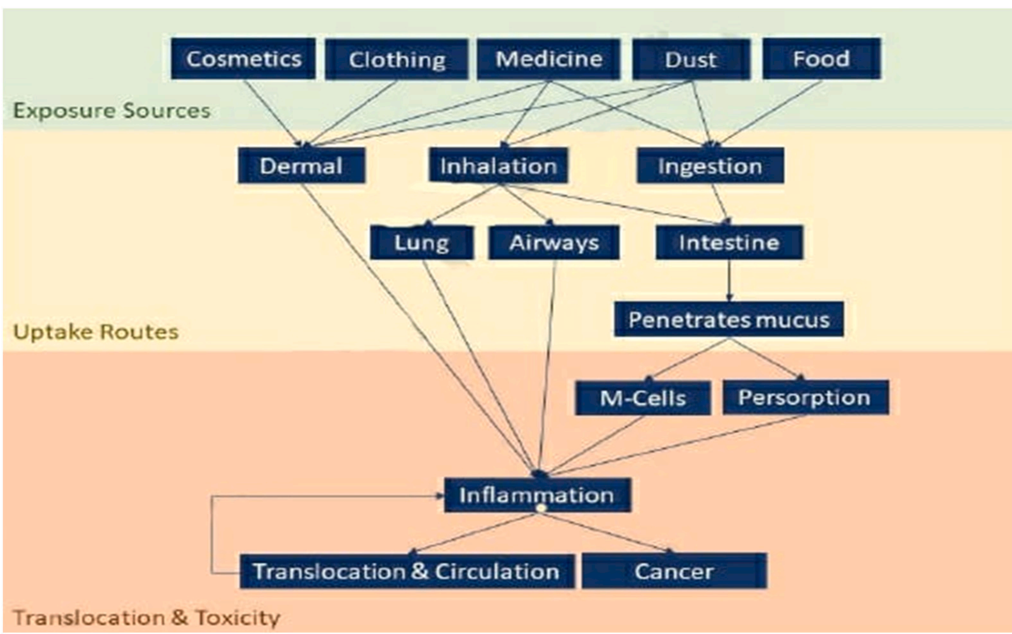

Fig. 2. Possible passageways of exposure and particle toxicity for MPs in the body.
Represented by the permission of Prata, Joana Correia, João P. da Costa, Isabel Lopes, Armando C. Duarte, and Teresa Rocha-Santos. "Environmental exposure to microplastics: An overview on possible human health effects." *Science of the Total Environment* 702 (2020): 134455.

forms such as rubber, wood, textiles, paper, plastic, etc., this only goes on to reiterate the dependence and prevalence of plastic. In the sense that the solids classified as readily degradable that can be seen in the form of paper, wood and natural fibers are easily degraded, but other materials that are termed non plastic, but still remain degradable such as ceramics from sea wrecks (Schleicher et al., 2008). People are open to MPs by breathing, intake and skin contact, finally causing the chronic prolonged inflammatory lesions (Fig. 2) (Prata et al., 2020).

The problem with plastic however is their non-biodegradable nature that exists in combination with their light weight, rendering them readily transportable by air and water currents. Among the research that goes into analysing the debris of the ocean, plastics have occupied a prime position, with wreckage investigation and fishery gear that became derelict in a close prominence. Previous studies have shown that among the debris collected from the surface, from the seabed and beaches. Plastics were found to be the greater fraction of floating debris in the ocean (Law et al., 2010), they are common in seabed samples (Galgani et al., 2000a) and, they were observed in large quantities during beach surveying and cleaning missions (Galgani et al., 2000b; Conservancy, Ocean, 2014).

A depiction of plastic as a serious threat to the marine environment can be traced back to the early publications of Marine debris prevalence (Ryan, 2015). Continued development of research into the problem of plastics reaching the marine ecosystem does not merely call for an assessment of the depth at which it impacts marine life and other linked ecosystems, but it also warrants the need to develop innovatory solutions in a rapid phase.

1.2. Microplastics: the smaller the size, the greater the threat

Plastics were thought to be the biggest threat posed to the marine environment until the discovery of MPs. In the beginning of this century, MPs were described as a collective debris of very minute or even microscopic plastic mass whose size is less than 5 mm (Andrady, 2011). Upon discovery, MPs became the greatest threat that we were faced with (Magnusson et al., 2016; Thompson et al., 2004). The definitive range of their size is debatable and it varies with every study, some estimating them at a diameter size < 1 mm (Browne et al., 2007; Browne et al., 2010a; Claessens et al., 2011a), while on the other hand, they have been linked to a much greater diameter of size < 10 mm (Graham and Thompson, 2009), and others with varying estimates in-between these ranges (Barnes et al., 2009; Betts, 2008; Derraik, 2002; Ryan et al., 2009).

A Research team from Korea observed the existence of MPs in 4

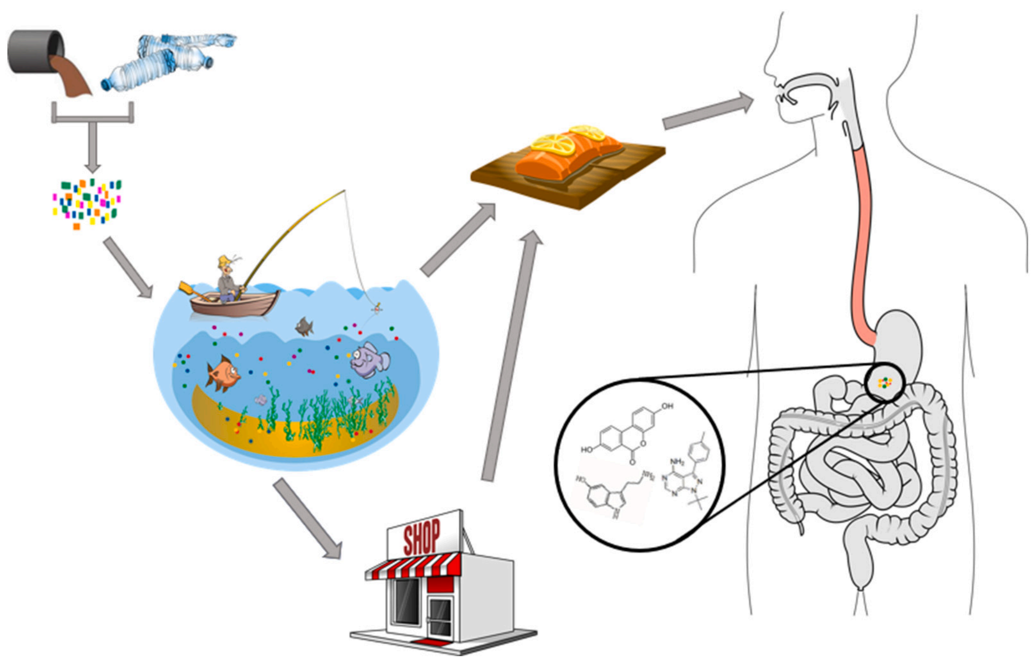

Fig. 3. A representation displaying how influence of human beings cause MPs to get in food network, make a way to our food and, finally, our organs.
Adapted from Cho, Youna, Won Joon Shim, Mi Jang, Gi Myung Han, and Sang Hee Hong. "Abundance and characteristics of microplastics in market bivalves from South Korea." *Environmental Pollution* 245 (2019): 1107–1116.

marketable bivalves from their 3 main cities, whose mean concentration of MPs was 0.15 ± 0.20 particles/g and it was assessed that the Korean people intakes 212 particles/person/year from shellfish ingestion (Cho et al., 2019). Entering of MPs in marine creatures is leading a conduit for litters and pollutants into our food (Fig. 3).

Such variations lacking consistency demands that a set of standards must be established in order to avoid problems that could potentially arise (Claessens et al., 2011b; Costa et al., 2010). There have also been suggestions of classifying a third kind of plastic based on size called the Mesoplastics, referring to MP debris that would be visible to the naked eye, but does not require the aid of a microscope (Andrady, 2011).

1.3. MP sources and its classification

MPs are broadly classified into primary and secondary MPs, based on their size. This is done based on the source of origin of such plastics. This classification seeks to differentiate the MPs that were manufactured to the current size from that which has undergone degradation to arrive at the current size. In the case of being manufactured to microscopic size, the debris thus created post application of the plastics, is termed primary MP debris, where in there was no need for the degradation of the plastic to attain its current size. Such plastics are often found in cosmetic products (Zitko and Hanlon, 1991), air blasting material (Gregory, 1996) and rarely seen applied in medicine (Patel et al., 2009).

Plastics of greater size such as pellets used for multiple household application that were suggested as mesoplastics were also seen to be a significant, yet subjectable addition to the contribution of primary MPs (Costa et al., 2010; Andrady, 2011). Cosmetic micro-scrubs were created as an exfoliating material, in competition against the traditional scrubs such as ground nuts, fibers and pumice (Derraik, 2002; Fendall and Sewell, 2009). Primary MPs have in addition, been found play a crucial role in air blasting technology, this technology removes rust and paint from a given industrial substrate that had undergone the deterioration process by using MPs like polyester(PES) (Browne et al., 2007; Derraik, 2002; Gregory, 1996). These air blasting scrubs are reused until they lose efficiency and at the point of discard, they also have heavy metal contaminants among the likes of lead and cadmium (Derraik, 2002; Gregory, 1996). MPs that has reached its classified size over degradation during a given period of time is called Secondary MPs (Magnusson et al., 2016; Thompson et al., 2004), which are degrade on land and reach the ocean in their designated size, or they are directly reach the ocean and degrade in it. Fragmentation of macroplastics (or mesoplastics) occur because of several factors such as physical, chemical, or biological (Browne et al., 2007).

Photo-oxidation of plastics by nonionizing rays such as UV rays, even from natural sunlight has been reported. These rays dissociate the polymer matrix by a bond cleavage (Browne et al., 2007; Halle et al., 2017; Hüffer et al., 2018; Moore, 2008; Rios et al., 2007). In order to tackle such oxidation reaction, additives are found to be used in most industries, resulting in a product cast with greater durability and resistance to photo-degradation (Talsness et al., 2009). Photo-degradation is not a concern to the plastic debris already in the ocean, as the marine aquatic conditions of temperature and salinity are not favourable for the photo-degradative process, but on land, plastics undergo a much more rapid process of photo-degradation (Barnes et al., 2009; Andrady, 2011; Moore, 2008).

Physical factors such as surface waves, turbulence of water currents etc., are also considered to be prominent factors driving fragmentation after the loss of structural integrity of the original debris that reached the ocean. This process is cyclic and will result in MP debris that is clearly classifiable as MP (Magnusson et al., 2016; Browne et al., 2007; Barnes et al., 2009; Fendall and Sewell, 2009; Rios et al., 2007). Other studies have suggested that these MPs do not stop their fragmentation at near micrometric scales but go on to form nanoplastics (Galgani et al., 2010).

2. Extraction of microplastics

2.1. Sampling methods

Broad observations of the debris collected from a given spot such as the surface of the ocean or the seabed could result in a misleading observation of various characteristics such as surface morphology and measures of particulate size. The area of collection could often be large as required by the nature of the research study. The extraction and separation processes of MPs is a laboratory based process that requires efficient and maximal isolation of MPs from the large samples that predominantly constitute masses that can infringe with further studies (Rocha-Santos and Duarte, 2015).

For the process of sampling, several plastics are used, these include vessels (Rocha-Santos and Duarte, 2015; Dubaish and Liebezeit, 2013), benthic trawls (Cole et al., 2011), bongo nets (Cole et al., 2011) and surface trawls (Lee et al., 2014). MP isolation from aquatic samples is easier, as compared to the soil and sediments from the marine environment. Samples from the general marine areas such as beach, estuaries and sea floor can also be used for MP isolation. This is done with the help of stainless-steel spatulas and spoons, if the sample is superficial, and the Cores and bottom trawls are used for deep sampling (Vianello et al., 2013a; Harrison et al., 2012; Cauwenberghe et al., 2013).

2.2. Extraction from sediments and waters

For the post sampling of water and sediments, the density separation techniques are applied to separate the MPs from the samples. High concentrates of salt are normally used to float a fraction of the MPs. The use of NaCl solution for such separations was first documented with samples collected from Norderney, a Northern Sea Island (Fries et al., 2013a). Following this, a density separation procedure based on NaCl solution was reported from Canterbury coast lines in New Zealand (Clunies-Ross et al., 2016). Other such ionic solutions have also been shown to be effective in this principle of gradient separation, some examples include, Sodium bromide, Sodium iodide, Zinc chloride and Zinc iodide. These solutions, however proved to be costly and toxic to the environment (Mintenig et al., 2017).

In line with expectations, the increase in gradient density of the solution, gave rise to the amount of MP recovered. NaI and $ZnBr_2$ were noted to have a significantly greater rate ($p < 0.001$) of recovery of MPs. The size of the particles being recovered has shown to be a strong factor, and had to be taken into consideration while determining the solution (Quinn et al., 2017). Prior treatment of the sample is reported to increase the amount of MP recovered from the sample, whereby stubborn debris such as algae and organic matter are removed. An additional step of peroxidation with H_2O_2 was found to increase the yield and remove debris with a negligible degree of destruction to the sample (Zhao et al., 2017). The use of the Fentons reagent causes very negligible damage to the intrinsic properties of the MPs and causes considerable reduction of the preparation time (Tagg et al., 2017). Ultrasonic extraction methods have also been demonstrated to be effective in retrieving MPs from the gastro-intestinal tract of fish, and the use of ultrasonication resulted in a reduction of hazardous occupational risks involved and raised protocol safety (Wagner et al., 2017).

For the preparation of samples, several devices have been developed in addition to the available methods of chemical extraction. Mechanical separation of MP particles from water was achieved and reported in 2016, wherein the separating device was constructed with pipes (PVC) and connectors(Fig. 4).

A disk was randomly drilled through its area with a mesh layer (1 mm and 50 μm) glued to it, that was designed for the process of separation. The lesser density of the MPs assured that they flowed to the top of the separator, with the water flow. This instrument gave a recovery rate of 97.25% (Wessel et al., 2016). Team from the Chinese Academy of Science had developed an integrative device that assured a comparable recovery of MPs from sedimentary samples (Zhang et al., 2015).

2.3. Extraction from organisms

Some of these sampling protocols are advantageous to the researcher and have a greater preference in the field than the others for the purpose of sample collection of MPs from sediments, waters, and biological samples. In the case of biological samples, pre-treatment is highly necessary, there must be a procedure that involves solutions such as H_2O_2 to discard the contaminating mass. Some of the other such pre-treatment agents include KOH, HNO_3, NaClO and HCl (Rocha-Santos and Duarte, 2015). Oxidation agents did not cause any significant damage to the isolated MPs, as seen at the point of observation, but these methods did take a toll on the degree of recovery of these plastics being extracted from the biological sample and its accuracy.

The use of enzymes as an extraction agent was found to be more suitable, effective in terms of both cost and time for biological samples for recovery with minimal destruction (Courtene-Jones et al., 2017). Several enzymes that are of digestive employability, such as proteases were used and optimized to suitable reaction conditions of effective degradation, bar the MP. The effect of such enzymes is also tested with MPs to understand any possibility of sample deterioration. Trypsin was found to be an effective enzyme, among those that were tested for the suitability as a pre-treatment agent. It showed one of the greatest degradation rates, with an 88% loss of extra biological material at a working concentration of 0.3125% of trypsin (Courtene-Jones et al., 2017). Several other methods have also been used as complexity of the biological samples increased.

One prominent method developed was to test and optimize the temperature at which the oxidizing agent degraded the most of extra biological matter in the sample. The treatment of the biological mass with KOH caused significant biological degradation at 40 °C, this temperature made the process time efficient, and inflicted very little damage to the MPs. Another good procedure was to pre-treat with NaI and then to treat with KOH as described earlier, which is also proved to be efficient (Karami et al., 2017). The addition of NaI solution was reported to remove any residual minerals that may have been persisting in the samples. MPs, regardless of type showed good degrees of retrieval and upon separation, were found to only have mild damage to characteristics such as color, weight and size, suggesting that these techniques have potential application prospects with biological samples (Roch and Brinker, 2017).

Determining suitable extraction methods becomes crucial for further studies. Variations in methods limits drawing comparisons, as there are no established standard protocols already available (Besley et al., 2017). Details of sampling, such as sampling depth, location of collection, extraction repeats and time for settling are parameters that are critical in line with literature. Trawl specifications, such as its texture and diameter are also essential to be studied for good isolation. Certain reports have emerged, suggesting that the season of the year also must be taken into consideration, while collecting the MP sediments. They observed some variations in the concentration of MPs with respect to season (Veerasingam et al., 2016). The presence of MPs within the environment of the lab may also cause great interference in analysis (Woodall et al., 2015). This goes on to demonstrate that development of procedures, and techniques are highly required to analyse MPs in aquatic environments. These standards must be globally agreed on and set up as keys to guided research, in order to meet its purpose (Woodall et al., 2015).

3. Identification and characterization

The presence of MPs is everywhere in the marine ecosystem, and its delirious impact on biological life forms were well understood, there arose a need to study these particles and the effects caused in detail with respect to their size (Lee et al., 2013; Canesi et al., 2015). These studies required a good understanding of the physiochemical properties of this particulate matter, thereby requiring a detailed characterization. A good characterization would further help to understand, the nature of these particles, such as their shapes, colors, and constituent polymer material. Here, we present a broad outline of prevalent characterization tools and their application in MPs characterization.

3.1. Optical and electron microscopy

Optical microscopy (Dissection microscopy) is a commonly used tool to study the larger particulate masses, ranging in at a size of about

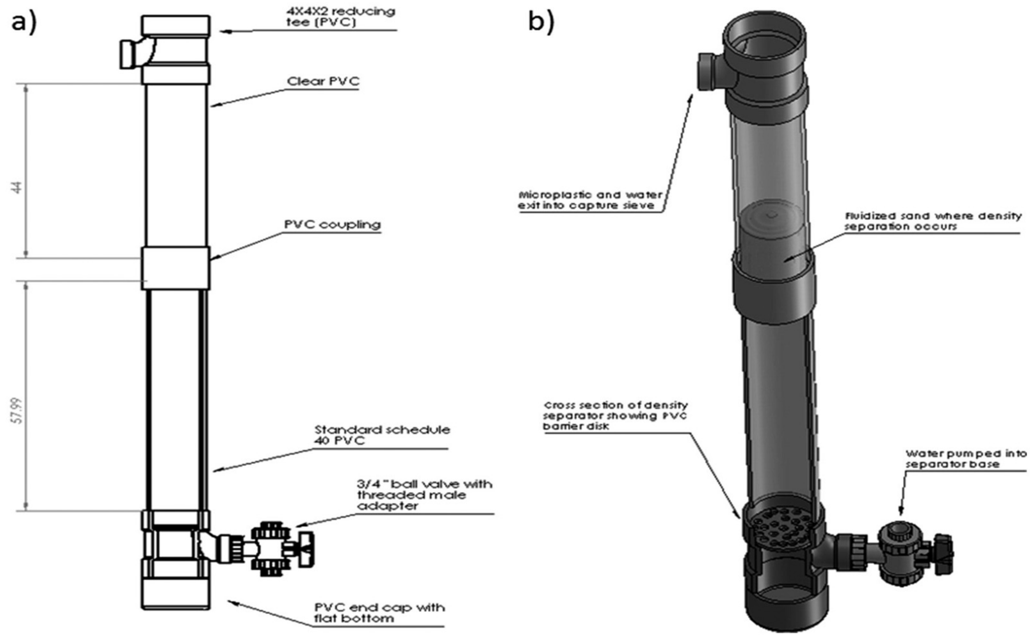

Fig. 4. Density separator design and setup. a) Part identification and assembly and b) functional depiction identifying internal components and separation process location.
Reprinted by the permission of Wessel, C.C., Lockridge, G.R., Battiste, D., & Cebrian, J. (2016). Abundance and characteristics of microplastics in beach sediments: insights into microplastic accumulation in northern Gulf of Mexico estuaries. Marine Pollution Bulletin, 109 (Resins, 2015), 178–183.

a 100 μm or more, as seen in the case of the net samples (Eriksen et al., 2014; Desforges et al., 2014; Laglbauer et al., 2014; Mathalon and Hill, 2014; Kang et al., 2015; Nel and Froneman, 2015). This method allows the study of surface texture and enables the differentiation of MPs from the contaminating ambiguous mass.

In spite of the most particles being easily identifiable under the optical microscope, there may be particles classified under the sub 100 μm range that can be very difficult to identify by optical microscopy, as in addition to their size constrain, they may also have no specified shape or color (Song et al., 2015). The contaminating sediment that persist because of poor separation in the gradient method of separation may also interfere during observation in MPs identification. Further, in extracting the biogenic material from sedimentary samples, the microscopic observation is difficult as a result of chemical digestion that has not been successful in eradicating the contaminants. Prior studies have also demonstrated that the false positive count was high for material being mistaken as MPs. It was seen that the average percentage for such misidentifications were high at the rate of 20% and in the case of transparent polymers, at 70% (65–67). This was later confirmed with spectroscopy. About 14% of these particles had a polymeric MPs composition, regardless of like resemblance (Löder and Gerdts, 2015). Microscopic methods also proved to be a weak means to distinguish between the synthetic and natural fibers (e.g., PES vs dyed cotton). Surveys have shown that fibers occupy a predominant position in the fractions of MPs found in the ocean, in the water, sedimentary and biological samples (Browne et al., 2010a, 2010b; Lusher et al., 2013).

The use of Scanning Electron Microscopy (SEM) can provide a much clearer image, given all these limitations of typical optical microscopy. The high-resolution nature of electron microscopy gives us a clear distinction between the organic particles and the plastic particles (Cooper and Corcoran, 2010). Further an Energy Dispersive X-Ray (EDX) analysis, can give us the exact elemental composition of the particles and ensure that the plastic particles are differentiated from the others as the plastics have a much greater percentage of carbon content (Vianello et al., 2013b).

3.2. Fourier transform infrared (FTIR) spectroscopy

Fourier Transform Infra-Red (FTIR) spectroscopy is another tool that is found to be greatly useful in the characterization of MPs. It gives the data on the available chemical functional groups in a given polymer. Every polymer produces a unique set of spectroscopic band signature that allows the differentiation and among the plastics, as well as of the plastics from the organic mass (Löder and Gerdts, 2015). A properly established and detailed database of available standard spectroscopic data for the various plastic polymers makes the identification of polymers an easy task. In the cases of very low particulate size of samples available, the option of micro FTIR (μ-FTIR) may be used (Song et al., 2014). In the μ-FTIR, the preliminary studies are conducted by switching between the objective lens and the IR probe prior to spectroscopic studies. Overview of the different analytical methods used to assess the concentration, chemical composition and morphology of MP's in biological tissues, sediments and water, from 2018 to 2020 is given in Table 1.

Phenomenon such as attenuated total internal reflectance (ATR), Transmission (Turner and Holmes, 2011; Ugolini et al., 2013), and reflectance (Ng and Obbard, 2006) modes are applied in the form of IR spectroscope operational modes for MPs analysis. As opposed to the transmission mode, the ATR and reflectance modes does not need any sample preparation step in the case of an opaque sample. Further, the ATR mode gives a stable and reliable spectral line data, even in the case of studying surfaces that have a rough texture, which would otherwise give out unstable spectral lines. It is understood that the particulates that have a size lower than the IR beam aperture are easily detectable by the probe.

3.3. Raman spectroscopy

Apart from the use of FT-IR, the use of Raman's spectroscopy for the identification of MPs are also a common practice (Van Cauwenberghe et al., 2013; Collard et al., 2015). Based on the molecular structure of the atoms on the surface, the laser beam that has been shot at the particles gives rise to a unique pattern of backscatter (Löder and Gerdts, 2015). The Raman's spectroscopy, in addition to identifying the plastic, it will also provide a composition of the polymers with respect to FTIR, which only allows an identification of the polymer. Further, in addition to the non-destructive methods of chemical analysis and microscopy, Raman's spectroscopy gives us a comparable tool of identification with the FTIR, bearing in mind the heavy cost of the instrumentation. FTIR and Raman spectroscopy can be used in a complimentary fashion with one another. The Raman spectroscopy methods allow the

Table 1
Overview of the different analytical methods used to assess the concentration, chemical composition and morphology of MP's in biological tissues, sediments and water, from 2018 to 2020.

MP type	Location	Environment matrix	Size of MPs	Concentration	Shape	Analysis method	Summary	Ref
PE, rayon	Victoria, Australia	Freshwater, P. australiensis (shrimp)	0.036 to 4.668 mm, 0.190 to 4.2 mm	0.4 ± 0.27 items/L, 0.52 ± 0.55 items/ind	Fibers	FTIR, SEM	Blue color and fiber-routine (in water and shrimp). common type-PE (water) and rayon (shrimp). primary studies of freshwater crustacean as a bio-indicator in measuring the MPs	(Nan et al., 2020)
PP, PE, Polystyrene (PS)	Three gorges reservoir, china	Surface water, sediments	G 1: < 0.5 mm G 2: 0.5–1 mm G 3: 1–2 mm G 4: 2–3 mm G 5: 3–4 mm G 6: 4–5 mm	1597 to 12,611 n/m^3, s 25 to 300 n/kg (ww)	Fibers, fragments, pellets, films, styrofoam	μ-raman spectroscopy	Water-PP,PE (abundant). Sediment-PS (abundant). Chemical pollutants were also identified. MPs sourcing in TGR is imperfect and further investigation is required	(Di and Wang, 2018)
Polypropylene (PP), PAN, PE, PA and PUR, polyester	Southern tyrrhenian sea	*Engraulis encrasicolus*, *Sardina pilchardus*	0.10–1.5 mm,0.25–0.83 mm,	0.26 items/specimen, 0.53 items/specimen	Fibers, fragments	Raman, FTIR spectroscopy, SEM	Presence of small amounts of organic dyes and components, (involved in the polymer matrix to adjust its base properties) on MPs.	(Savoca et al., 2020)
PVC, Polyamide, PE	Irish waters	Crustacean *Nephrops norvegicus*	1–2 mm	1.75 ± 2.01 items	Fibers	FTIR	Positive connection among prawn caraspace condition and MPs abundance. 15 to 4471 particles/year of seafood for human intake	(Hara et al., 2020)
PS foam, PP	El Quetzalito sand beach, Guatemalan Caribbean	Sand Beach	1–5 mm	279 items/m^2	Fragments, beads	Mid-infrared spectroscopy	PS, PP are common in trade, profitable, fishing, and domestic activities which state the necessity to develop proper ecological management for solid waste treatment and discarding	(Mazariegos-Ortíz et al., 2020)
PS, PP and PE	Southern Caspian sea	Surface water, sediments	–	34,490 particles per km^2, 210 particles per kg	Fragments, foams	ATR-FTIR, Optical microscope, X-ray (EDS)	The capability of MP pollution and the need to determine the level of pollution in southern caspian sea	(Mataji et al., 2020)
PE, PS, nylon	Andaman and nicobar islands	Water Sediment Zooplankton Finfish Shell fishes	35.29 to 5010 μm	0.93 ± 0.59 particles/m^3, 45.17 ± 25.23 particles/kg, 0.12 ± 0.07 pieces/zooplankter and 10.65 ± 7.83 particles/specimen	Fibers, fragments, pellets	Stereomicroscope, ATR-FTIR	Results show important relations amid fish weight and length with the consumed MPs in the fish gut, MP ingestion – Large creatures > small ones. The MPs residing duration in the larger organisms GI tract may be greater than the small organisms	(Goswami et al., 2020)
PE, PP, PL, PA, PS, PUR, PVC	Southwest coast, kerala	Coastal waters, beach sediments, marine fishes	Coastal waters: 0.3–0.6 (41%), 0.6–1.18 (22); 1.18–2.36 (19) and 2.36–4.75 mm (18) Sediments: 0.3–0.6 (44%), 0.6–1.18 (21); 1.18–2.36 (21) and 2.36–4.75 mm (21)	Coastal waters: 1.25 ± 0.88 particles/m^3, beach sediments: 40.7 ± 33.2 particles/m^2	fragments, fiber/line and foam	ATR-FTIR, FP-XRF	Wide number of metalloids, other elements and heavy metals that are indicating dangerous chemicals exist in MPs. Improved the idea on the sources, transfer pathways and the related environmental threats	(Robin et al., 2020)
PA, PE, PP	Brisbane river, Australia	sediments	< 3 mm	0.18 to 129.20 mg/kg, or 10 to 520 items/kg.	Fiber, fragment, film	ATR-FTIR	Believes that river sediments acts as a sink for plastic accumulation. It emphasizes that an established standard	(Durukan and Karadagli, 2019)

(continued on next page)

Table 1 (*continued*)

MP type	Location	Environment matrix	Size of MPs	Concentration	Shape	Analysis method	Summary	Ref
							unit should precede to develop relation among studies and to avoid over- or under-estimation of the extent of MP pollution in aquatic system	
PE, PVC, PP	Atlantic coast, France	*Mytilus edulis* (Blue mussels)	30 to 200 μm	–	fibers	μ-FTIR Microscopy	Great retrieval amounts were achieved particularly for PE and PVC (80% recoveries). This procedure permitted to notice and find MPs (20 to 100 μm)	(Phuong et al., 2018)
PE, PP	China	Tap water	< 50 μm	440 ± 275 particles L-1	Fragments, spheres, fibers	Micro-raman	Consumable water treatment units have to face the issue of MPs contamination in tap water because of eco-toxicological effects on human beings.	(Tong et al., 2020)
PS, PE	Caspian sea, north of Iran	Sediments	250–500 μm	25 and 330 items/kg	Film, fragment, fiber	SEM-EDS analysis, PLM and Raman micro-spectroscopy	MPs distribution showed an uneven spatial pattern suggesting that the greater MPs quantity are near permanent rivers and in the regions with increased range in fishing and tourism events	(Mehdinia et al., 2020)
PP,PE, Polyesters, PET, PVC	Rawal lake, Pakistan	Surface water, sediments	≤ 1 mm	Water: 0.142 items/0.1 L, sediments: 1.04 items/0.01 kg	Fibers, fragments	FTIR	Higher conc. of MPs in the sediments in comparison to Rawal Lake water. Increased population density surrounding lake, inappropriate garbage disposal, tourism, and leisure time activities may be the major reasons for the lake's MP pollution	(Irfan et al., 2020)
Polyester, PP, PE, Polyacrylate, PS, PU, PDMS	Municipal waste water treatment plant, Thailand	Water	–	Influent: 12.2 pieces/L, aeration tank: 138.2 pieces/L, effluent: 2.0 pieces/L	Fibers, Fragments, spheres	FTIR	Advised to start tertiary treatment to develop MPs removal in WWTPs, and to put on post-treatment to the WWTPs' raw slurry to prevent the MPs' release into the environment when the slurry is smeared to farming land	(Hongprasith et al., 2020)
PP, PS, PC, PE, PET	Yellow sea and bohai sea, China	Neophocaena asiaeorientalis sunameri(7 East asian finless porpoises)	–	19.1 ± 7.2 items/individual	Fibers, fragments, foams	Raman	Trophic transfer and ingestion (unintentional) might be the potential pathways for MPs intaken by finless porpoise. Intestinal structure(specific one) might account for the predominance of fibers and the accumulation of MPs at the starting part of intestines	(Xiong et al., 2018)
GPPS, PE, PET, PA, PP	Coastal areas of Negros oriental, Philippines	*Siganus fuscescens* (rabbitfish)	1–5 mm	30mp per site	Fibers,	FTIR	MPs were absent in the guts of fish, likely due to the different location and character of their feeding habitats.	(Bucol et al., 2020)
PE, PP, rayon, acrylic	Korea	Meretrix lusoria(clam), Scapharca broughtonii	100–300 μm(43%), 1000–3000 μm (37%), 1–5 mm(16%)	Clam: 0.08items/g, blood arkshell: 0.05items/g,	Fibers, fragment	Light microscope, FTIR	MPs composition was mostly rayon, semi-synthetic cellulosic	(Borkar et al., 2020)

(*continued on next page*)

Table 1 (*continued*)

MP type	Location	Environment matrix	Size of MPs	Concentration	Shape	Analysis method	Summary	Ref
		(blood arkshell), styela clava(warty sea quirt)		warty sea quirt: 0.12 items/g			material and polyester, which are main component of fabric and textile	
PE, PP, PS, PVC, PET,ABS, PEUT, CA	Hong Kong	Sediments	0.5–5 mm	100pieces of different size, color, shape	Fragment, fiber, foam, pigment, film	FTIR	Mechanisms behind adsorption and desorption processes research between metals and MPs will aid in calculating the risks to both human health and the environment.	(Li et al., 2020)
PP	Tambak Lorok coastal area, Sem arang, Indonesia	Sediments, sea water	Sediments: 255–6137 μm, sea water: 270–1279 μm	Sea water: 7–111 particles/10 mL, sediments: 8–49 particles/g	Filamentous, fibers	FTIR	At the surface of seawater, PP underwent photo-oxidation degradation at 50 cm depth and biodegradation at 170 cm.	(Khoironi et al., 2020)
PS, PP, PE	15 stations, Persian gulf	Surface water	1000–3000 μm (32%), 500–1000 μm (29.2%), 3000–5000 μm (22.5%), 100–500 μm (16.4%)	899 total counted particles	Fibers, fragments, film, pellets	ATR-FTIR	Collected MPs has mainly PE, blue and white color. Parameters like population, sewage and wastewaters inputs, shipping, fishing and industrial activity in the coasts influence the MPs abundance	(Kor and Mehdinia, 2020)
PE, PET, rayon	Qingdao, China	Tap water, Jihongtan Reservoir water, Laoshan Reservoir	10 to 5000 μm	Tap water: 0.3 to 1.6 items/L, reservoir sources: 0.2 to 0.7 items/L	fibers	ATR- μ-FTIR	Great risk of the MPs was evaluated by the polymers potential risk index. Outcome showed that water treatment decreased the MPs dangers in tap water.	(Zhang et al., 2020)
PET, PE, regenerated cellulose	New Zealand	sediments	< 300 μm (4) 300–500 μm (39) 500–1000 μm (35) 1000–5000 μm (21)	459 particles.m^{-2} ranging from 0 to 2615 particles.m^{-2}	Fibers, fragments, films	ATR-FTIR	Great variability seen between sites with no link detected between spatial data, signifying that MPs richness is very site-specific.	(Bridson et al., 2020)
ABS,CA, EVA, PC, PE, PETE, PMMA, P, PS, PTFE, PU, PVC	Urban coastal environment, suva, Fiji	sw, sediments and fish gastrointestinal tracts of the fish	–	–	Fibers, fragments, microbeads, films	ATR-FTIR	The sewage treatment plant indicated to give to MP levels in sediment only, not in water. Fragments and fibers were the major MP type, and increased quantity of PE, PP and latex were obtained	(Ferreira et al., 2020)
PA,PU/PET blend, Acrylonitrile-acrylate, PS	Gulf of Tigullio, NW Italy	Sediment of torrents and sea bottom	–	Torrents: 1.5 items cm^{-3} ± 1.3 SD, Sea bottom: 1.6 items cm^{-3} ± 1.3 SD	Fibers, fragments, pellets, films, granules	FTIR spectroscopy 2D imaging	Tuning the methods and spectral analysis used for μl sampling and analysis is important to facilitate in contrast with the results reported by other studies	(Cutroneo et al., 2020)
PE, PS, Silicone, PTFE, Polyester, rayon	South Korea	See the foot note[a]	< 1 mm – 90% 1-5 mm – 10%	See the foot note[b]	Fibers, fragments	FTIR	Habitat occupancy is also important in evaluating the MPs abundance in fish than feeding habit. PTFE's density cause the ingestion by bottom-dwelling.	(Park et al., 2020)

[a] Surface water of the Han river and its tributaries, carp (*C. carpio*), crucian carp (*C. cuvieri*), bluegill (*L. macrochirus*), bass (*M. salmoides*), catfish (S. asotus), and snakehead (*C. argus*).

[b] Surface waters 1. At 0 m was 0–42.9 particles/m^3. 2. At a depth of 2 m was 20.0–180.0 particles/m^3. River tributaries: 1.2 to 234.5 particles/m^3. Intestines of fish: 4 to 48 particles/fish. Gills of fish: 1 to 16 particles/fish.

Table 2
Sampling procedure, Pre-treatment for analytical method like Py-GC/MS used for characterization and determination of MPs in water, fish, and sand from 2019 to 2020.

MP type	Location	Sampling procedure	Pre-treatment	Size	Summary	Remarks	Ref
PE,PP PET,PVC, PS or PA	Boknafjorden, South-west Norway	Wet residues collected at a site-eight kilograms, refined, separated them according to size using 10-250 mm mesh size sieves	Focused onto filters of fiberglass	10–40 µm	Shifts which are observed in size distribution were also in some sites and were related to the bottom currents of marine sea and the impact of some anthropogenic activities.	Good sensitivity, reliability, and rapidity.	(Gomiero et al., 2019)
PS	Italy	Diff size (3 m and 45 m) by lamella, branchia and stomach related organs – displayed for four days at diff conc polymer	Tissues of mussels(bivalves) were cured with 10% KOH at 900 °C with satisfying assimilation effectiveness(97 + 1%)	2–10 m	Pyrolysis results showed that the concurrent importance of particle size and mass concentration suggests the need to include both variables in ecological threat investigation	May hamper the detection of styrene oligomers. Last pyrolysis item is inclined to framework obstructions	(Fabbri et al., 2020)
PS, PS, PVC, PET	Atlantic and Mediterranean marine	Refer the footnote[a]	Sifted on a glass fiber channel (15 mm, 1 m pore 7 size; pre-rewarded in a muffle furnace at 400c for twelve hours	< 1 mm	Patterns (regional one's) of MP components are distinguished and allow mapping for more targeted source associated research. FdS MPs reveals the MP mass load of related coastal waters and runs its chronological/geographical studies	Has a great capability to make MP mass parts from compound ecological sample	(Fischer et al., 2019)
PE, PP	France	1.5 kg of dry residues - acquired. They were sieved with a 5, 2, and 1 mm sieves	Flotation treatment	–	Impact of tide lines was revealed only on particle abundance at Boulogne-surMer, might be because of black pellets(high proportion) at this tide line.	Slight estimation of 80% pyrogram is important to ensure appropriate recognizable proof	(Périne et al., 2019)
PE, PS	Germany	filters with mesh sizes of 100 mm, 50 mm and 10 mm were used	–	25,70 and 120 µm	Polymer mass quantification for detected polymer types was questionable for Σ-GC/MS, whereas further methods were unsuccessful to define the correct polymer mass.	Minimal sample preparation with short analysis times	(Müller et al., 2020)
PE, PP, PS	Germany	Methanolic pre-removal was implemented to minimise alarming effects of the matrix. Removal of MPs was allowed with THF at 185 °C and hundred bar.	–	10–50 µm and 200–400 µm	Solid samples in particular, the analysis of triplicate analysis revealed that the raised statistical uncertainties because of non-uniform MPs distribution. Care to be taken because agglomerates might form during milling and homogenization of samples	Enables low quantification limits when combined with pressurized liquid extraction	(Dierkes et al., 2019)
PE, PP, PS, PVC, PA, EPDM	Brazil	Gathering residues, drying, filtering by using 0.25,0.5, 1.0, 2.0 mm sizes of mesh	–	0.1–1 mm	Approximate total MPs profusion and distribution at the sieve meshes tested, calculate thread/fiber and fragment proportions, and segregate MP samples for the following Py-GC/MS study to recognize their compositions.	Refer the foot note[d]	(Gimiliani et al., 2020)
PE, PP, PS	German river elbe	Apstein mesh of plankton (opening: 0.022 m^2, Ø 17 cm, size: 110 cm, mesh extent: 150 µm)	Refer the foot note[b]	125–5000 µm	PE and PP, being routine polymer kinds in the water phase, whereas highly diverse polymer distribution was witnessed in the case of sediments	Refer the foot note[c]	(Scherer et al., 2020)

(continued on next page)

Table 2 (*continued*)

MP type	Location	Sampling procedure	Pre-treatment	Size	Summary	Remarks	Ref
PET	Italy	1 kg residue gathered from 10 cm top, filtered to eliminate > 2 mm fragments, and precisely homogenized before taking 3 subsamples, about 15 g each, for triplicate test	–	1–5 mm	Presence of great range of PET MPs in sediment brings the necessity to explore more regarding the presence and extent of contamination	Good accuracy and sensitivity. No data on molecule and size, cross-obstructions with pyrolysis items causes under or over-estimations.	(Castelvetro et al., 2020)
PE,PET, PVC, PS, PAA, EVA, Polyamide-12	Cape town, South Africa	Samples were collected by 300 µm neuston net, cleaning and stockpiled in 1 L glass bottles	Flotation treatment	–	MPs showed varied surface morphological patterns with the inorganic elements such as Cl, Na, Ca, Al, Si and Fe	Proper technique for subjective determination of the pyrolysis items	(Vilakati et al., 2020)

[a] Sample to a sieve of 20 m and washed with pre-separated (1 m) faucet water. The non-dissolvable particles were moved with NaBr arrangement from sieve into a 200 ml division channel, vivaciously shaken. 40 min – allowing to settle, bottom particles discarded.
[b] Powders of ground filters were re-suspended in H_2O, permit elements to settling down and filtered the supernatant(2×). Suspended channel powder was totally moved on another channel and allowed to dry for 7 days at 55 °C.
[c] Higher analytical sensitivity. Results showed that sampled MP with PS reference has PS was crosslinked with divinylbenzene might had impact on identification of styrene by Py-GC/MS technique.
[d] Financially savvy, creates less natural effect than those as of now accessible. Successful in blocking the requirement for solvents and decreasing the preparation time of sample.

characterization of particles ranging in size at the level of few microns, this is made possible by the very narrow slit beam in the Raman Spectroscope (Cole et al., 2013).

Raman spectroscopy is advantageous in the sense that it is, like FTIR, a noncontact method. This is further used to identify MPs among zooplankton samples, which is made possible by the confocal microscopic attachment seen in the Raman Spectroscopy (Cole et al., 2013).

On the contrary, Raman's spectroscopy had the great disadvantage in Interference faced by the additives and pigments to make the final plastic cast meet the requirement (Van Cauwenberghe et al., 2013; Tagg et al., 2015). List of studies carried out on using Py-GC/MS for the past 2 years are given in Table 2.

3.4. Thermal analysis

Among the tools used in the identification of MPs, the thermoanalytical method is the most recent tool to make debut, where in it is used to study changes in the intrinsic physiochemical properties of the plastic with respect to its thermal stability (Tagg et al., 2015; Castañeda et al., 2014).

One such tool is the Differential Scanning Calorimetry (DSC), which studies the thermal properties of the unknown polymer microparticles (Tsukame et al., 1997). This technique requires the use of reference materials for the identification and matching of a given MP sample. Therefore, this technique is prevalently used in the identification of primary plastics, which readily have reference material such as micro beads of PE (Castañeda et al., 2014). The idea of attaching thermogravimetric analysis (TGA) to DSC was tried, and it was observed that this could help to differentiate between the PP and PE polymers, but the method faced the problem of overlap in phase transition and as a result could not be able to identify few important polymers such as PVC, PES, PA and PET (Majewsky et al., 2016).

TGA in combination with solid phase extraction (SPE), and being coupled to a thermal desorption gas chromatography mass spectrophotometry (TDS-GC–MS), grants the user a set of advantages. It allows larger sampling size in comparison to a Py-GC/MS and grants greater resolution when compared to a DSC (Dümichen et al., 2015). TGA-SPE-TDS-GCMS was found to be effective in the identification and quantification of PE from a sample of soil and mussels, whereas the PP, PS and mixed polymer also gave out similar results to validate this method (Dümichen et al., 2015).

Py-GC/MS is the most commonly used tool for identification of the polymeric type today. In the Py-GC/MS technique, the polymer is pyrolyzed under inert atmosphere, which was then fed to a gas chromatography (GC) coupled with mass spectrometry, in which GC separates the pyrolyzed products and pyrogram is generated. The pyrogram of the unknown samples are compared with available or developed reference pyrogram to understand the constitution of the polymer mass under study. The method allows the use of relatively much lesser mass, in the range of 0.35–7 mg of particulate debris, at temperatures as high as 700 °C in comparison with TGA. The bulk of the analysed sediments and solid particulates under suspension revealed the presence of PVC, PS, poly(vinyl acetate) (PVA) and styrene-butadiene styrene rubber in good resolution (Fabbri et al., 2000; Fabbri, 2001). Py-GC/MS was also used to study the particles such as PA and chlorinated polyethylene (CPE)/chloro-sulphonated polyethylene (CSPE) (Fries et al., 2013b; Nuelle et al., 2014; Dekiff et al., 2014). According to the instrument condition, we need to choose the pyrolyzing filament for the identification of the polymer.

4. Future directions

The characterization of MPs by thermal techniques studied so far and to be applied in future are highlighted in Fig. 5 below. In the existing literature, studies on MPs thermal degradation are usually reported using Py-GC/MS technique and thermal desorption gas

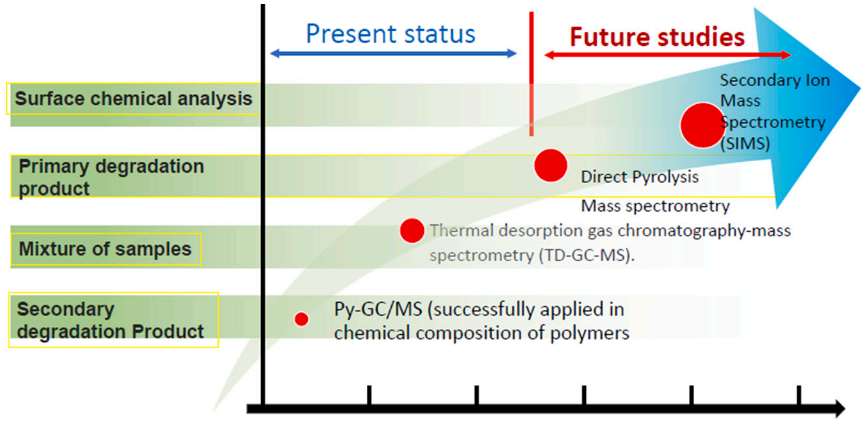

Fig. 5. Characterization of MPs by thermal techniques.

Table 3
Comparison between DPMS and Py-GC/MS.

Pyrolysis	DPMS	Py-GC/MS
Residence time in pyrolysis zone	Less than a second	Milli seconds
Pyrolysis products	Polymer is pyrolyzed very close to the ion source, and the primary chain cleavage products are instantaneously reached to the detector. Primary pyrolysis products are detected	Primary pyrolyzed products have enough residence time to go through secondary reactions. Secondary pyrolysis products are mostly detected
Thermally labile products	Can be detected without any secondary reactions	Secondary reactions are possible
Molecular weight effect	Higher molecular weight degradation products can be analysed	Generally, higher molecular weight degradation products are lost in the column or after formation

chromatography–mass spectrometry (TD-GC–MS), which could be due to the expertise only in these techniques. The characterization of MPs by direct pyrolysis mass spectrometry (DPMS) and secondary ion Mass spectrometry (SIMS) have to be studied in future.

Earlier studies on the analysis of the degradation products of MPs using the Py-GC/MS technique provided information only on the secondary degradation products. The MPs by both DPMS and Py-GC/MS techniques, will have to be studied in future, which will shed light both on the end groups that are formed during the hydrolysis and/or photodegradation (please note photodegradation does not happen for sample in ocean/water) of these MPs in marine environment. Generally, DPMS technique has been applied to study the degradation products of most of the polymers, to cite a few hydroxyl terminated polybutadiene (HTPB) (Ganesh et al., 2000), polysulfides (PLS) (Sundarrajan et al., 2002; Sundarrajan et al., 2005; Montaudo et al., 1994), PET (Montaudo et al., 1993) and so on. In DPMS technique, polymer is pyrolyzed very near to the ion source, and the primary chain cleavage products formed are instantaneously reached to detector for obtaining the mass spectra. The comparison between DPMS and Py-GC/MS technique are briefly presented in Table 3. In future, we are aiming to study the MPs in marine environment by using the above two techniques. As the time scales of the two pyrolysis techniques are very different, we expect that thermally labile pyrolysis products, end group formed during photo-oxidation of MPs in the sea-shore and hydrolysed groups formed in the case of MPs in marine (aqueous) environment will be detected in DPMS. Also, a comparative study by DPMS and Py-GC/MS may be able to detect different chemical compounds.

Detection limit of MPs in the marine environment must be improved, which can enter into human food chain through fish. Secondary ion mass spectrometry (SIMS) has been widely used to characterize the polymers, additives in polymers, and so on. However, to the best of our knowledge (confirmed by Sci-finder search), it has not been applied to characterize the MPs in marine environment. This study will provide an insight into the functional groups formed through the hydrolysis and/or photo-degradation of these MPs in marine environment and thereby its human health impact can be assessed, which has also to be studied in future. It is to be noted here that only two reports are available on SIMS, in which 1) metal ion diffusion into plastics (Kern et al., n.d.) and 2) sea surface exposure (Jungnickel et al., 2016) are studied.

5. Conclusion

There is much left to study about these MPs debris that are making a great hindrance for the Marine eco-system. While we are currently capable of understanding the individual composition, there are several limitations to these tools, such as the reduced size, of these particles that sometimes falls beyond the frame of the characterization or isolation method, the time consuming extraction processes and persistent non-plastic mass. The development in available technology seen today for the purpose of isolation and identification of MPs is a result of slow improvement that happened over four decades to facilitate the rise of demand for efficiency and speed in this process. With the understanding of the need to study this debris being consistent, there is a strong need for better and advanced technology to aid the researcher. In conclusion, we suggest that further advancements developed must take into consideration, the fragmenting nature of this debris and seek to reduce the minimal separable and identifiable size of the MPs. Earlier studies on the analysis of the degradation products of MPs using the Py-GC/MS technique provided information only on the secondary degradation products. The identification and characterization of MPs by both DPMS and Py-GC/MS techniques, will have to be studied in future, which will shed light both on the end groups that are formed during the hydrolysis and/or degradation of these MPs in marine environment. In addition, SIMS studies must be carried out, which will shed light on formed functional groups, adsorbed metal ions, and other adsorbed species on MP surfaces.

CRediT authorship contribution statement

The manuscript, figure and tables were contributed by Kavya. Reviewing, write up and improving manuscript quality, Future directions, literature search partly, abstract and conclusion part and subsequent corrections were performed by Subramanian Sundarrajan. The overall direction and guidance were provided by Seeram Ramakrishna.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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