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Progress on methods used for detection, characterization and removal of microplastics from marine environment: A mini review

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Abstract

Microplastics (MPs), small sized plastic particles of <5 mm, are ubiquitous in marine environment. MPs pollution in marine water due to anthropogenic activities is a global concern. In recent years, adverse effects of MPs on human and aquatic life forms were reported. It is highly important to obtain a comprehensive understanding of MPs and its impact on environment and human health. Identification and separation of MPs from marine environment is highly challenging due to their complex physicochemical properties. This mini review elaborates on the emerging techniques used for characterization of MPs and the advancements in the removal methods.

Keywords: Characterization, Detection, Marine pollution, Microplastics, Removal

1. Introduction

Over the past five decades, plastics have become an integral part of our life due to their diverse applications. Properties of plastics such as light weight, long life and high hydrophobicity have made them an attractive material for packaging of products [1]. The demand for plastics increased from 270 million tonnes per year in 2016 [2] to 370 million tonnes in 2019 [3]. Unfortunately, this exponential growth in plastic usage has also manifested itself through an ever-increasing presence of plastics in waste streams, both on land and in water [4]. Among a variety of debris found in marine waters, plastic wastes occupy the prime position. Marine waters are considered to be the largest sink of plastic pollution on Earth, carrying over 5.25 trillion micro and nanoplastics [5]. Non-biodegradability and buoyant nature of plastics makes them easily spread across the marine environment [6]. Plastics can undergo complex transformation due to atmospheric changes, interaction with sunlight, environmental matrices, and ageing [7].

Based on the size, plastics are categorized as macroplastics (>25 mm), mesoplastics (5-25 mm), large microplastics (1-5 mm), small microplastics (<1 mm), and nanoplastics (1-1000 nm) [8]. Among these, microplastics (MPs) received much attention in recent years, because of the fact that MPs are found even in the food and the air [9]. MPs are often categorized as primary MPs and secondary MPs based on their origin and evolution. Primary MPs are of very small beads of polymers, microfibers, and pellets which are mainly found in cosmetic products, clothing etc. Secondary MPs are degradation products of larger plastic particles such as soda bottles, plastic bags, fishing nets etc. [10]. Most abundant MPs in marine water are polyethylene (PE), polyethylene terephthalate (PET), poly vinyl chloride (PVC), polypropylene (PP), cellulose acetate (CA), polystyrene (PS) [11]. Around 80% of these MPs have come through land-based sources such as landfills, run off from agricultural lands, industries, household activities and degradation of parts of ships [12]. Health care products such as toothpastes, cosmetics, facial scrubbers, bath foam, are also sources of MPs which can enter collectively as large quantity into the sewage system eventually entering sea [13]. Improper methods of recycling plastics is yet another potential source of MPs. MPs in farm lands are due to usage of greenhouse materials, manure, pipes used for irrigation, and so on which eventually enters marine water through run offs. As a whole, the level of MPs pollution in marine water is taking greater heights every year forming a potential threat to human and marine ecosystem. MPs originating from various sources eventually make their way to marine environment. This review aims to give an overview of the recent progress made in the detection, characterization, and removal of MPs from the marine environment.

2. Detection and characterization of MPs

MPs exhibit diversity in shapes, size, color etc. Identification and characterization of MPs are essential to determine their concentration, chemical composition, and its effect on various life forms. Detection, sampling, and handling techniques depend upon the size of MPs. These processes are difficult and demanding for very small MPs, with a high probability of producing false positive and negative results. General analytical techniques involve identification (detection), extraction, isolation (separation), characterization and quantification. Analytical methods exclusive to characterization are still in a developing stage. A combination of physical and chemical analyses is widely employed for its characterization. Table 1 gives the list of techniques used for detection and characterization of MPs in marine water and their limitations. Most commonly employed techniques in the analysis of MPs are discussed here.

2.1 Visual inspection

Large MPs of size greater than 500 µm can be detected visually by naked eye. MPs can be segregated based on size, shape, color, and type of plastic after which they are counted. This method is subjective, may produce variations between observers. For example, the count of MPs may vary according to individual, experience, fatigue, resulting in overestimation or underestimation.

2.2 Chemical methods

Color can be used for the identification of chemical composition and source. Use of dyes can assist the ease of identification. Dyes such as Oil red EGN, Eosin B, Hostasol Yellow 3G and Rose Bengal have been used [14]. Dye Nile Red have been very promising. Staining with Nile Red requires short incubation time and allows characterization using vibrational spectroscopy. Materials of bio-origin such as wood, feathers, shells etc. are not stained by Nile Red [15].

Table 1 Analytical methods used for the characterization of MPs.

Technique	Principle	Advantage	Disadvantage
Optical detection	MP's present in the sample is manually counted by naked eye or with the help the microscope	Cheap	 Time consuming Possibility of over estimation or under estimation Depends on the observer
FT IR spectroscopy	 Infrared radiation interacts with the sample, stretching or bending vibrations which results in a change in dipole moment absorb radiation Existing data libraries can be used for analysis. A minimal amount of samples is needed. No sample preparation. Can detect small particles (~25 μm). Can be used for studying weathering of samples 		 Expensive instruments used. Can take considerable time for the whole particle identification. Additives or contaminants can overlap with the signals
Raman spectroscopy	Scattered light is used to measure the vibrational energy modes of a sample.	 Less sample requirements No sample preparation is normally needed. Can detect small particles (1-2 μm). 	 Expensive instruments. Time consuming and laborious work. Overlapping of signals due to the presence of additives or other contaminants.
Microscopy	A microscope is used to analyse substances. Optical microscopes to electron microscopes such as SEM and TEM are widely used.	High resolution images	Requires laborious preparation steps. Extremely time consuming
Thermal analysis	Physical and chemical properties of a substance is analysed by using heat-based techniques	No sample preparation is normally needed. A large number of samples can be identified at once	Time consuming Quantitative analysis of each sample is not possible

2.3 Raman microscopy

Raman microscopy is widely used for the characterization of MPs of size $< 20 \, \mu m$, mainly due to high spatial resolution [16]. Microscopic pieces of plastics are analysed by focusing a laser beam onto a small spot to obtain Raman spectra. Plastics, made of polymers, have a unique set of vibrational modes which can be used for chemical identification. Characterization of marine MPs is highly challenging due to various physical and chemical transformations such as oxidation, breakdown, biofouling, weathering etc. For instance, the PE MPs in ocean were characterized and the microstructure was analysed using Raman microscopy. Results show that PE MPs got weathered and underwent oxidation resulting in lowering of molecular weight compared to the non-weathered objects. Adsorption of trace metals and organic pollutants were also detected on the outer layer [17].

Micron sized PVC particles are one of the common pollutants in marine water. Raman imaging of micro and nanoplastics which are generated from PVC pipe cuttings were reported. They discovered that a single cutting process of PVC pipe generated thousands of MPs in the range of 0.1-5 mm, millions of small MPs in the range of 1-100 μ m and billions of nanoplastics in the range of <1 μ m [18]. Combining Raman spectroscopy with other techniques could be advantageous to identify the type of MPs. Raman spectroscopy coupled with multivariate analysis was used for analyzing MPs samples from mineral plastic boxes, water bottles and medicine vials. Sensitivity, specificity, and accuracy of the analysis was found to be more than 99% [19].

2.4 Infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) is widely used for analysing MPs of size <100µm. Changes in the dipole moment of a bond due to molecular vibrations, results in infrared absorption. Infrared spectroscopy can be used for identification of functional groups present on polymers. For example, infrared (IR) peaks used for the characterization of most common polymers are PE: 718 g/cm (CH₂ rocking), PP: 2839 g/cm (CH₂ stretching), PET: 1724 g/cm (C=O stretching), Polyamide (PA): 3300 cm⁻¹ (N-H stretching) [20].

Harsh conditions are prevalent in marine environment and the polymers can undergo surface degradation under sunlight which can be monitored by FTIR. On weathering, new chemical bonds such as carbonyl groups, hydroxyl, and carbonoxygen are formed. Carbonyl index (CI), defined as the absorbance of carbonyl bond peaks relative to the absorbance of reference peaks (methylene bond) is commonly used to measure the light induced photo-oxidation of polyethylene MPs. The carbonyl absorbs in the region of 1760–1690 g/cm and the methylene scissoring absorption bands happens in the region of 1490–1420 g/cm [21].

Low density polymers float on the surface of ocean and sea for long period of time. Ageing of these polymers, undergoes transformation on the surface from smooth to rough and due to surface oxidation of aged polymer such as low-density polyethylene (LDPE), a new and stronger band at 1706 g/cm ($\nu_{C=O}$) was found, confirming the formation of carbonyl. Weathered polystyrene MPs showed new bands at 3360–3240 g/cm (O-H stretching), 1640 g/cm (C=O stretching), and 1100 g/cm (C-O stretching) [22]. Availability of various modes such as transmission, reflectance and attenuated total reflectance (ATR) is another attractive feature of infrared spectroscopy. ATR-FTIR can be used for the characterization of large MPs (5mm-500 μ m) and smaller MPs (< 500 μ m) by μ -FTIR [23].

Raman micro spectroscopy (RMS) and μ -FTIR together can be used for analyzing MPs and it can even produce unambiguous results with significant accuracy. A manual analysis, semi-automated method, and fully automatic identification method of μ -FTIR and Raman techniques for analysis of MPs were compared and semi-automated method was recommended because, manual method was time consuming and fully automated method gave many false positives and negatives [24].

2.5 Thermal analysis

Thermal analysis includes different analytical techniques such as thermogravimetry (TGA), pyrolysis gas chromatography mass spectrometry (Py-GC-MS), TGA-MS, TGA thermal desorption GC MS (TGA-(TD)-GC-MS) and TGA-differential scanning calorimetry (TGA-DSC). In Py-GC-MS, polymer sample is heated under inert conditions and decomposition products are separated by GC and analysed by MS. Py-GC-MS method was found to be advantageous when compared with conventional solvent extraction methods as it was cost effective, fully automated and less time consuming. Both polymer and organic plastic additive contents can also be analysed in a single run [24].

Characterization of MPs based on thermal profile alone can be ambiguous. For example, the DSC profiles of PE and PP was found to be endothermic and peak temperatures were different. Whereas, for other polymers DSC profiles were similar. Hyphenated techniques always produce better and more accurate results. Thus, TGA combined with FTIR was used for analysing Polystyrene (PS) and PVC MPs for the first time [25]. Semicrystalline (PP, PA, High density Polyethylene (HDPE), LDPE and PET) and amorphous polymers (PS and PVC) were characterized by combining TGA and DSC [26]. Both amorphous polymers were identified by DSC using the glass transition temperature. Semicrystalline polymers were identified by the melting temperature without the interference of other polymers. DSC coupled with optical microscopy were used for identification and quantification of MPs made of LDPE, HDPE, PP and PET [27].

2.6 Scanning electron microscopy

Scanning electron microscopy (SEM) is an important tool to study the morphology, ageing and origin of the MPs by examining the surface features such as grooves, pits, fractures, and flakes on the MPs. Modern instruments have an Energy Dispersive Spectroscopy (EDS) detector along with SEM which can do elemental analysis based on detecting the characteristic x-rays emitted. SEM-EDS gives both qualitative and quantitative information on the composition of the sample. It can be used for distinguishing organic (rich in Ca/Mg/Sr), inorganic (minerals, salts) and MPs (rich in C/Cl/S/Ti) [28]. Although SEM is a good technique to analyse MPs, the technique is time consuming mainly for sample prepreparation and observation and it cannot be used for large number of MPs.

3. Quantification of MPs

To reduce MPs entering marine waters it is highly important to remove them at their origin. Wastewater is a significant contributor of MPs into marine ecosystem. Hence, removing MPs from wastewater could significantly reduce the concentration of MPs entering the marine environment. Hence it is crucial to look into the improvements of removal methods used in wastewater treatment plants (WWTPs). In WWTPs, almost 75% of MPs are removed in the primary stage of treatment. Due to the very small size of MPs, the majority of them escape the secondary stage. It is in the tertiary stage, almost 75-98% of MPs are removed [29]. Recent advancements in various methods adopted for the removal of MPs from wastewater are discussed in detail. Table 2 provides a summary of the advantages and limitations of the methods discussed here.

Table 2 Removal of microplastics by various methods.

Method	Principal	Advantages	Limitations
Density separation	Difference in densities	Low costEase of operation	High density polymers cannot be removed
Sand filtration	Polluted water is distributed in the top of the sand filter and will slowly sink through the sand filter bed.	Ease of operationUsed as primary treatment process	Not efficient
Membrane technology	Membrane filters	Removes MPsLow filtration resistance	Sludge formationNeeds frequent cleaning
Electrocoagulation	Electrochemistry	Ease of operationRemoves small MPs	Supply of ElectricityChange of electrodes
Magnetic method	Hydrophobic attraction	Cost effective.Small size MPs can be removed	Can produce more secondary MPs

3.1 Density separation

Density separation is one of the most commonly used techniques to separate MPs from the environment based on the difference in specific density of sediments and plastics. Saturated salt solutions with a defined density are used for this process. Most commonly used salt solutions are saturated sodium chloride (NaCl), zinc chloride (ZnCl₂), sodium iodide (NaI), zinc bromide (ZnBr₂) and sodium polytungstate (3Na₂WO₄·9WO₃·H₂O). NaCl is commonly used because of its availability, cheap and harmless nature. It is highly efficient in extracting low density MPs such as PE, PP and PS and less efficient for high density MPs such as PVC and PET [30]. NaI, ZnCl₂, ZnBr₂ and sodium polytungstate can efficiently remove MPs with high density [31].

ZnCl₂ gave better results when compared to NaCl, NaI and NaBr. ZnCl₂ is toxic to aquatic life forms, expensive and some are even found to have long lasting effects [32]. Recycling and reuse of ZnCl₂ and NaI solutions make the process more cost effective and minimize environmental pollution. The methodology for reusing ZnCl₂ involves the mixing of ZnCl₂ solution to the sample. MPs are separated and recovered based on difference in density and ZnCl₂ solution is vacuum filtered and reused. The results from this study reveal that ZnCl₂ solution can be reused for upto five times retaining an efficiency greater than 95% with MPs [33].

On the other hand, NaI is unstable due to the complex equilibrium exhibited by sodium and iodide resulting in change in the chemical behavior of NaI. A small decrease in the density of NaI is also observed, simultaneously affecting the extraction efficiency of MPs. To address this, studies on different storage conditions of NaI and its impact on density were carried out. It has been observed that NaI solutions can be recycled with just 4.8% loss of initial mass without any changes

in the density of the solutions [34]. In 2021, potassium carbonate (K₂CO₃) with a density of 1.54 g/cm³ was reported to remove PVC particles with removal efficiency of ~90% [35].

Density separation is usually carried out in glass beakers, funnels, upside funnels which has various drawbacks such as time consumption, slow recovery and so on. A small glass device without a vlave is designed for rapid separation of MPs from sediments [36]. With this simple device, they have shown a recovery of 94–98% of MPs with a size range of <1,000 µm. A novel flotation separation method for removal of PVC plastics was reported by increasing the hydrophilicity by selective deposition of ferric ions. By optimizing the ferric ion concentration, the wettability of PVCs can be adjusted thereby separating them by flotation [37]. The densities of various salts that are commonly used for the studies for removing MPs of various types and their limitations are given in Table 3 and Table 4 summarizes the densities of separated polymers in g/cm^3 .

Table 3 Extraction of microplastics by density separation using solutions of various densities [41].

Solutions Used	Density (g/cm ³)	Separated Polymers	Limitations
NaCl	1.17	PP, LDPE, PS, Nylon, PE	Cannot extract high density microplastics.
NaBr	1.37	PP, LDPE, PS, Nylon, PE, HDPE, PVC, PET	Expensive and hazardous
Sodium polytungstate (3Na ₂ WO ₄ ·9WO ₃ . H ₂ O)	1.4	PA, PE, HDPE, PET, PP	Causes serious eye damage; harmful to aquatic life with long lasting effects
$ZnCl_2$	1.46	PP, PS, PVC	Hazardous to aquatic environment
NaI	1.56	PP, LDPE, PS, Nylon, PE, HDPE, PVC, PET	Expensive and hazardous
$ZnBr_2$	1.71	PA, PE, PVC, HDPE, PET, PP	Expensive and hazardous

Table 4 Densities of various polymers.

Polymer type	Density (g/cm ³)	
Polyethylene (PE)	0.865 - 0.912	
Low density polyethylene	0.91 - 0.925	
High density polyethylene	0.959 - 0.965	
Polypropylene (PP)	0.91 - 0.928	
Polystyrene (PS)	0.96 - 1.05	
Polyvinyl Chloride (PVC)	1.38	
Polyethylene Terephthalate (PET)	1.38	
Nylon	1.13-1.15	

3.2 Sand filtration

Rapid Sand filtration (RSF) technique is a method used to remove MPs originating from sewage and drinking water. Artificial samples from plastics bags and tyre flakes of different sizes (10 - 500 μ m) were taken for the study and MPs of size greater than 200 μ m were removed [38]. The removal efficiency of the MPs by sand filtration was 29.0-44.4% compared to coagulation/sedimentation. Two different membrane bioreactors (MBR) and RSF were tested for the removal of MPs found in wastewater treatment plant. The removal efficiency was found to be ~79% for MBR and ~75% for RSF [39]. Aluminosilicates modified by cationic surfactant was used as a filter for the removal of frequently found MPs such as granular PE MPs (~10 μ m) and fibrous PA MPs (~100 μ m). A significant removal efficiency of >96% was achieved when compared to RSF (63%). The retention mechanism of MPs involves capture, trap and entanglement which was confirmed by SEM images [40].

3.3 Membrane separation

Membrane separation is a very simple and energy efficient process in removal of MPs when compared to conventional techniques. Various Membrane processes include microfiltration, ultrafiltration (UF), nanofiltration and reverse osmosis based on the pore size of the membrane used in the treatment process. UF membranes having a pore size of 1-100 nm are

used to remove MPs and organic matter present in wastewater in combination with coagulation. The efficiency of this process is considerably less for small size MPs [41]. Instead, nanofiltration can help to remove MPs of very small size.

Membrane based technology was already extensively applied in the removal of suspended particles, bacteria, dissolved gases, hardness causing ions and salinity in water. It has gained attention towards combating MPs pollution in water bodies. A series of biomimetic gill-inspired membranes (BGIMs) with micropores of size 3.5-10.5 μm, was observed to be efficient for the removal of MPs from polluted water [42].

PC, CA, and polytetrafluoroethylene (PTFE) membranes with pore size of 5 μ m were used for the removal of PA and PS MPs of size 20-300 μ m with high efficiency. The difference in the performance of the membranes is attributed to the varying nature of interaction with the membranes. Large size MPs (20 μ m) break down into small size (5 μ m) and pass through the membrane which are 5 μ m in diameter. Comparing the mass removal efficiency of the membranes, CA was performing better for long term [43].

3.4 Electrocoagulation

Electrocoagulation (EC) makes use of two metal electrodes (anode and cathode), dipped in a container with wastewater. In the presence of electric field, anode undergoes oxidation producing metal cations. On the surface of cathode, water reacts to form H_2 gas and hydroxyl ions. These metal cations and hydroxide ions react to form metal hydroxide micro coagulants. The MP pollutants and the coagulants collide with each other to form flocs which settle down and then removed by filtration.

MPs such as PE, PMMA, CA and PP were removed by electrocoagulation method using Al and Fe as anode. The results illustrate that the performance of Al anode is better than Fe. The mechanism of removal involves the formation of Al^{3+} and Fe^{3+} ions by dissolution. With the increase in pH, Al^{3+} and Fe^{3+} react with OH^- generated at the cathode. Metal cations can undergo hydrolysis when it comes in contact with water [44]. Al^{3+} hydrolyses form complex species such as $Al(H_2O)_5OH^{2+}$, $Al(H_2O)_4OH^{2+}$, $Al(H_2O)_3OH^{2+}$, etc. and they further undergo hydrolysis to form polynuclear species which ultimately transforms into amorphous flocculant ($[Al(OH)_3]_n$). The MPs are then removed by adsorption which happens due to electrostatic attraction of charges on the surface of MPs and polynuclear hydrolysed species (Figure 1) [45]. Due to high removal efficiency, electrocoagulation method of extracting MPs is considered as a preferable choice among various treatment methods reported so far [46].

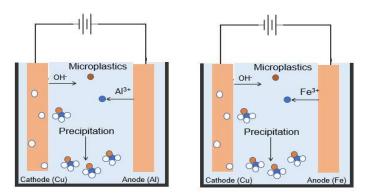


Figure 1 Schematics illustrating the mechanism of MPs removal from water by electrocoagulation using Al and Fe as coagulating agent [45].

3.5 Magnetic separation

The smaller the size of MPs, the greater the threat and damage to the environment mainly due to their high surface to volume ratio. Moreover, they carry other organic pollutants and contribute to biomagnification in marine ecosystems. Extraction of MPs of size <150 µm are difficult and challenging.

Separation of small sized MPs using magnetic materials has received a lot of interest. Surface hydrophobicity of MPs helps in the removal of MPs by hydrophobic-hydrophobic attraction mechanism. Recovery of MPs were achieved by using Fe nanoparticles modified with hexadecyltrimethoxysilane (HDTMS) offering hydrophobic nature to the surface by the formation of siloxane bonds (-Si-O-Si-). These magnetic nanoparticles attract MPs by hydrophobic attraction and removed by external magnetic field. Three different size range of MPs were tested, ranging from large (\sim 1 to \sim 8 mm), medium (200 μ m to 1 mm) and small size (<20 μ m). By this method, around 92 % of small size MPs was recovered [47].

A magnetic carbon nanotube (M-CNT) was prepared for removal of MPs. Three different types of MPs were considered for the study such as PE, PET and PA of diameter 48 µm. The mechanism of extraction is based on the adsorption of PE by strong hydrophobic interaction and adsorption of PA is mainly due to hydrogen bond formation. In the case of PET,

there is π - π interaction along with hydrophobic interaction. Maximum adsorption was observed in neutral pH and the final agglomerates were removed from the system within 300 minutes [48].

Ferrofluids without stabilizing agents or surfactants can also be used for MPs removal. Ferrofluid is prepared by mixing oil and iron oxide (Fe₃O₄) powder. The experimental conditions were optimized by varying the volume of oil and quantity of iron oxide powder at a ratio of 1:2.5 (volume of oil: dosage of magnetite). Lubricating oil successfully removed 99% of MPs from water [49] (Figure 2).

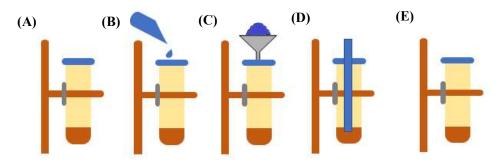


Figure 2 The process of MPs removal using ferrofluid by mixing oil and iron oxide powder [49]; (A) microplastic suspension, (B) 2.5 mL/L oil added, (C) 9.5 mL/L of magnetite powder added and shake well, (D) insertion of magnet to trap ferrofluid, and (E) Remained water sample analyzed.

Attempts were made to remove MPs using nano-Fe₃O₄. Surface of the MPs were magnetized with nano-Fe₃O₄ resulting in the change in the morphology. MPs which were originally smooth, white, and transparent changed to brownish yellow. MPs (PE, PP, PS and PET) treated with 1.3 gL⁻¹ nano-Fe₃O₄ for 150 minutes caused significant absorption. More than 80% of MPs can be removed from environmental water bodies using this method [50].

A magnetic nanocomposite based on a polyoxometalate ionic liquid (POM-IL) and core-shell Fe₂O₃/SiO₂ nanoparticles was reported found capable of removing organic, inorganic, microbial and MPs pollutants. POM-IL coated on magnetic nanoparticles gets attached to MPs which was then magnetically recovered [51].

4. Conclusion

MPs are one of the major contributors of global marine and environmental pollution. MPs of different sizes, shapes etc. are abundant and widespread across the marine environment. The source and composition of MPs in marine environment is well understood, but efficient and advanced tools are needed to facilitate the researcher to analyze MPs. As the size of MPs becomes smaller, analysis of MPs is challenging. Use of a single technique may not be sufficient in detection and characterization of MPs, and hyphenated techniques are considered to be of great help in analyzing MPs. MPs undergo degradation and ageing due to interaction with marine environment. Advanced techniques are needed for monitoring the degradation pathway of MPs. In situ detection of MPs in marine environment is at an infant stage. A standard protocol has to be developed for analyzing and removal of MPs in marine waters.

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