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Detection and Characterization of Microplastics in Drinking Water Using Advanced Analytical Techniques

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KEYWORDS

Microplastics, Membrane filter, FTIR, Scanning electron microscopy, XRD

ABSTRACT:

Introduction: The universal problem of microplastic pollution has drawn significant global attention in recent years. The detection of these tiny plastic particles in drinking water raises concerns about possible health hazards and emphasizes the need for a thorough investigation into their origins, distribution, and effects on human health.

Objective: The present study aimed to detect the presence of microplastics in drinking water from various sources including bottled water from 2 branded companies and one local brand and tap water.

Methods: By using field water testing kit developed by the Tamilnadu Water Supply and Drainage Board (TWAD) the water samples were analyzed for parameters such as pH, dissolved oxygen (DO), turbidity, conductivity, temperature, phosphate, chlorine, ammonia, nitrate, fluoride, and iron. Additionally, the water samples were filtered through membrane filtration techniques to detect the microplastics in the water samples. The membrane was further analyzed for the presence of microplastic using FTIR (Fourier Transform Infrared Spectroscopy), XRD (X-ray Diffraction), and SEM (Scanning Electron Microscopy)

Results: FTIR results revealed the presence of some contaminants in the water samples. SEM analysis revealed particle size of 2µm which may confirm the presence of microplastics in the water samples. The XRD pattern exhibited an amorphous or semi-crystalline peak, further indicating that microplastics may be present. It is suggested that microplastics may have been introduced into the water samples during processing.

1.INTRODUCTION

The synthetic, organic polymer known as plastic is derived from fossil fuels like petroleum and gas. The United Nations Environment Programme reports that more than 460 million metric tons of plastic are generated annually. Single-use items including straws, shopping bags, cigarettes, bottles, caps, and cups are the main source of plastic pollution worldwide (UNEP). Greenhouse gasses and other pollutants, such as carbon dioxide, dioxins, and methane, are released into the atmosphere when plastic garbage is burned (IUCN).

Global plastic production reached 367 million metric tons in 2020, generating 29.1 million tonnes of waste plastic. Of this, only 9% of plastic garbage is recycled, 12% is burned, and the remaining portion is either landfilled or disposed of outside [3]. Numerous

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industries, including necessities, agriculture, and the military, heavily rely on plastics. Significant amounts of unprocessed plastic garbage have entered the environment as a result of the extensive use of plastic items [4].The widespread occurrence of plastic pollution in our waterways, dumps, and even within our own bodies could be viewed as an important indicator of the Anthropocene [5]. Terms like “our plastic era” [6], “plastiglomerate” [7,8] “anthropoquinas” [9] and “the plastisphere” [10] have emerged.

Plastics vary in size and shape and are classified based on size into categories such as megaplastics (length > 50 cm), macroplastics (5–50 cm), mesoplastics (5 mm–5 cm), microplastics (MPs) (1 µm–5 mm), and nanoplastics (<1 µm) [11].

Microplastics are classified into two main categories based on their source as primary and secondary. Primary microplastics are intentionally produced, entering the environment through sewage and wastewater systems, whereas secondary microplastics result from the fragmentation of larger plastic waste via physical, chemical, and biological processes[12]. The most common shapes of microplastics include fragments, granules, fibers, and films[13,14,15]. Microplastics are primarily composed of polymers like polyethylene terephthalate (PET), polyethylene (PE), and polypropylene (PP). Although these plastics were designed to be chemically inert and pose minimal risks to human health, the environmental and biological implications of their widespread presence are concerning [16].

Microplastics are now widely detected in both terrestrial and aquatic ecosystems such as groundwater, wastewater, surface waterbodies, and marine waters. It is reported that eighty to ninety percent of microplastics in water bodies originate from land-based sources [17]. Remaining 10–20% of the microplastics introduced via maritime activities by tourists, commercial fishing, marine boats, and offshore operations [18].

Microplastics (MPs) have been detected in organisms at every level of the food chain, indicating their potential to bioaccumulate and move up trophic levels. There are three possible ways that these tiny particles can enter the human body: by eating, airborne exposure, and cutaneous contact. skin lesions may make it easier for tiny particles to enter through syringes or catheters. Environmentalists have been raising concerns about plastic pollutions possible effects on the environment and, more recently, on human health for more than 50 years [19].

Swan and Colino (2021), in their book Count Down, claim that constant exposure to endocrine disrupters such as Bisphenol A and phthalates found in plastic is accountable for increased infertility problems and obesity rate. Studies show that annual human consumption of microplastics, depending on age and sex, ranges from 39,000 to 52,000 particles [20]. Most evidence about the toxicity of MPs comes from animal models and in vitro studies on human cells, if these particles accumulate it may potential health risks in humans [21,22]. To enhance plastic durability or performance, additives are added that may be associated with toxicity [23]. Once ingested or inhaled, microplastics can accumulate in tissues and bodily fluids, leading to inflammation and other adverse biological effects [24].

Scientists have discovered plastic particles in the deep ocean [25], in rainwater [26], on Mt Everest [27], in human placenta [28] and blood [29], in the water we consume, in planktons [30]. A recent review also emphasizes that plastics are certainly leading to environmental impacts, but the degree of harm resulting from microplastics is significantly more intricate to evaluate [31].

Numerous studies have shown that microplastics can be found in drinking water sources worldwide, such as tap water, bottled water, and groundwater. This study aims to investigate the presence of microplastics in locally sourced and branded water bottles by employing analytical techniques such as FTIR, XRD and SEM.

2.MATERIALS AND METHODS

Sample Collection:

Branded and local water bottles were collected from local market in Ambattur, Chennai. Using a sterile container, tap water was collected from residential area in Krishnapuram, Ambattur. Water samples were analyzed using field water testing kit developed by TWAD board (Tamilnadu Water Supply and Drainage Board) for the presence of Nitrite, Chlorine, Phosphate, Fluoride, Iron, Ammonia, Nitrate**.**

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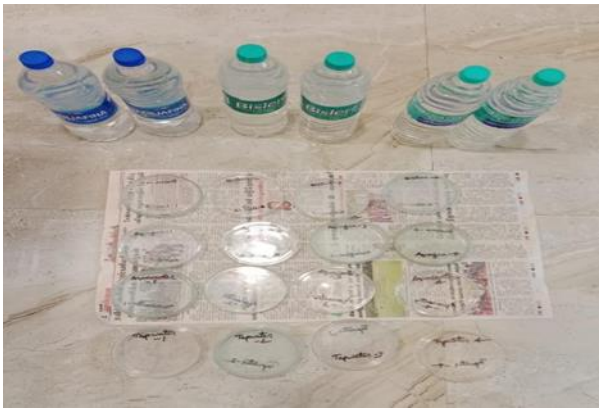

Fig 1: Water samples

Membrane filtration

Membrane filtration is commonly used to detect microplastics in drinking water by separating particles based on their sizes through a filter membrane. Smaller particles and dissolved materials pass through the membrane, while microplastics larger than the pore size are trapped in the membrane.

Fig 2: Membrane filtration

The membrane holding the trapped microplastics is carefully removed, subjected to spectroscopic analysis using methods like Fourier-transform infrared spectroscopy (FTIR), Xray diffraction and Scanning electron microscope.

Fourier Transform Infrared Spectroscopy

Water samples were characterized for the presence of functional groups using FTIR spectroscopy. The dried filters containing the particulate matter were directly placed on the ATR (Attenuated Total Reflectance) crystal. Spectra were recorded in the wavelength range of 4000–400 cm^{-1} with a resolution of $4cm^{-1}$. The obtained spectra were analyzed to identify characteristic peaks corresponding to different polymer types. The peaks in FTIR spectra, measured in wavenumbers (cm^{-1}), indicate the absorption or emission of infrared radiation at frequencies. A specific molecular vibration, such as the stretching, bending, or twisting of chemical bonds, is represented by each peak [32].

Scanning Electron Microscopy

Analyzing microplastics, in water samples using Scanning Electron Microscopy (SEM) involves directing an electron beam over the materials surface. This technique reveals detailed information about the surface morphology of microplastics. The equipment consists of an electron gun as the electron source and a set of lenses to propel and accurately concentrate the electron beam. Scattered electrons are captured by detectors positioned at angles to produce images displaying the surface properties of the microplastics[32].

X-Ray Diffraction (XRD)

XRD analysis was performed using a diffractometer. The dried samples were placed on the sample holder, and diffraction patterns were obtained using Cu Kα radiation (λ = 1.5406 Å) over a 2θ range. The resulting diffractograms were analysed to identify crystalline phases present in the samples

RESULTS AND DISCUSSION

The physico chemical parameters of water samples along with BIS standard is shown in table 1. Colour in bottled water samples were of 5 Hazen units referring to water as white and tap water is slightly yellow colour with Hazen unit of 8. Odour and taste were agreeable. The pH range according to BIS standard is 6.5-8.5. The water samples pH ranged from 6.8-7.4. This shows that the water samples were slightly alkaline.

The nitrite concentration in all the water samples were found to be 0.2 mg/l but it is found within the permissible limit. Nitrites can be harmful at higher concentrations. Nitrate levels were consistent across the branded water samples (20 mg/l), whereas tap water exhibited a higher concentration of 45 mg/l. High nitrate levels in water can cause methemoglobinemia or blue baby syndrome, a

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condition found especially in infants less than six months old.

Chlorine was undetectable in all water samples except tapwater, suggesting that either no chlorine was added, or the residual chlorine levels were below detectable limits. The amount of free chlorine in tap water was found to be 0.2 mg/l, the presence of chlorine may be due to tertiary treatment of drinking water. Liquid chlorine is mixed into drinking water to destroy bacteria.

Phosphate was detected in brand 1 and Tapwater samples (0.5 mg/l) , while brand 2, local brand had no detectable levels. The fluoride levels in all samples were within safe limits for consumption (0.5-1 mg/l). Dental fluorosis is the most frequently observed effect of fluoride exposure. Skeletal fluorosis characterizes the most severe negative health effect that is linked to over exposure to elevated levels of fluoride in drinking water.

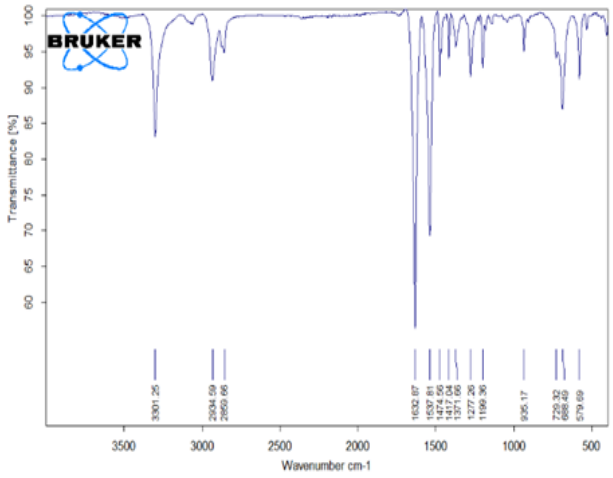
There was no Iron found in the water samples suggesting that the levels were below detection limit.

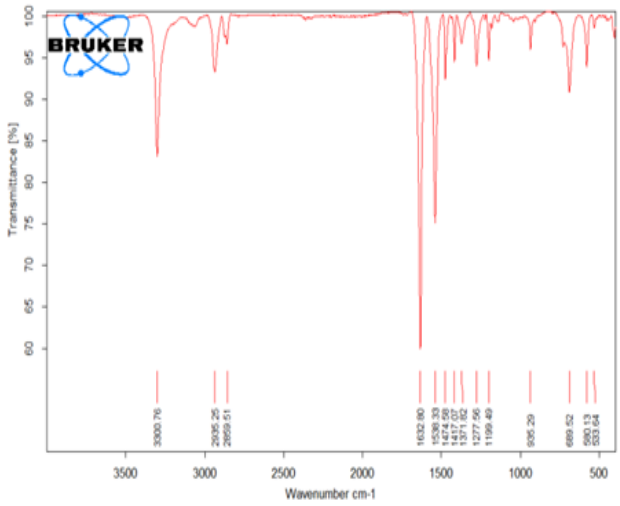
The concentration of ammonia in brand 1 and tap water sample was found to be 1.0 mg/l and 2.0 mg/l, respectively, while brand 2 and local brand had no detectable ammonia. The higher ammonia level in tap water could indicate contamination from organic matter or sewage, raising concerns about water quality. Ammonia has a toxic effect on healthy humans only if the intake becomes higher than the capacity to detoxify [33].

Water samples were filtered through membrane filter and due to high amount of TDS in tap water,it was cumbersome to filter tap water. So in further studies the tap water sample was avoided.

Table 1: physicochemical parameters of water samples

	BIS standards ISO 10500 : 2012	Brand 1	Brand 2	Local Brand	Tapwater
Colour (Hazen units)	5	5	5	5	8
Odour	Agreeable	Agreeable	Agreeable	Agreeable	Agreeable
Taste	Agreeable	Agreeable	Agreeable	Agreeable	Agreeable
Ph	6.5-8.5	7.4	6.8	7.3	7.1
Nitrite (mg/l)	2	0.2	0.2	0.2	0.2
Nitrate(mg/l)	45	20	20	20	40
Free Chlorine(mg/l)	0.2	0.0	0.0	0.0	0.2
Phosphate(mg/l)	5	0.5	0.0	0.5	0.5
Fluoride(mg/l)	1.0	0.5	0.5	0.5	1.0
Iron(mg/l)	0.3	0.0	0.0	0.0	0.0
Ammonia(mg/l)	0.5	0.0	0.0	0.5	2.0





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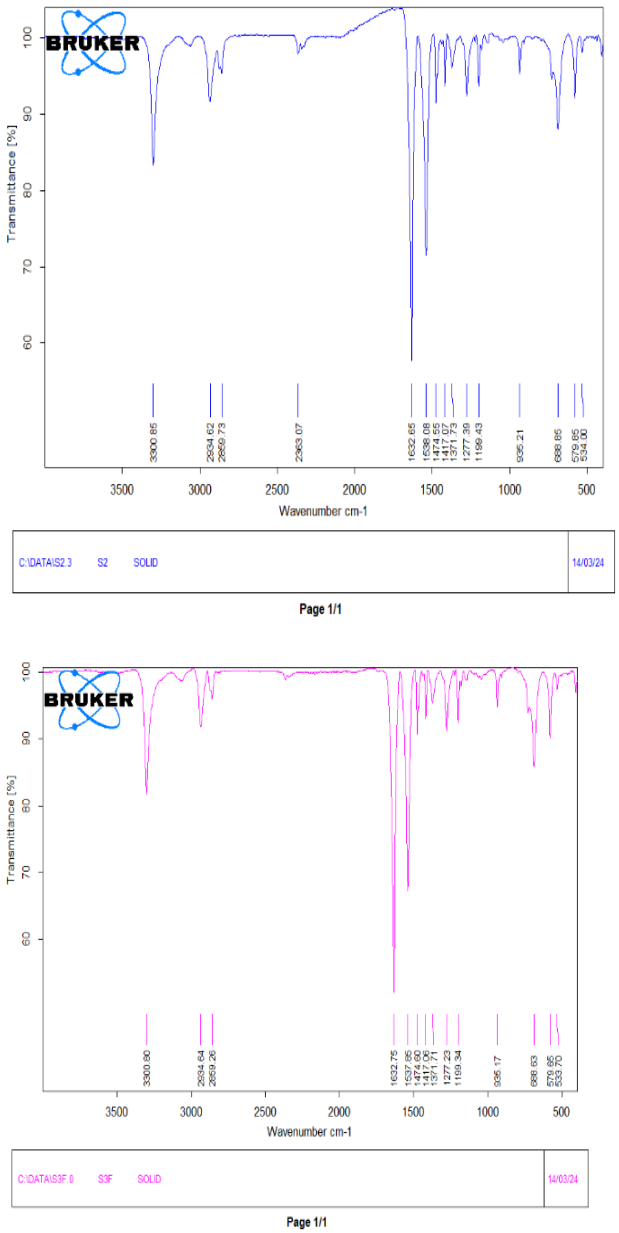
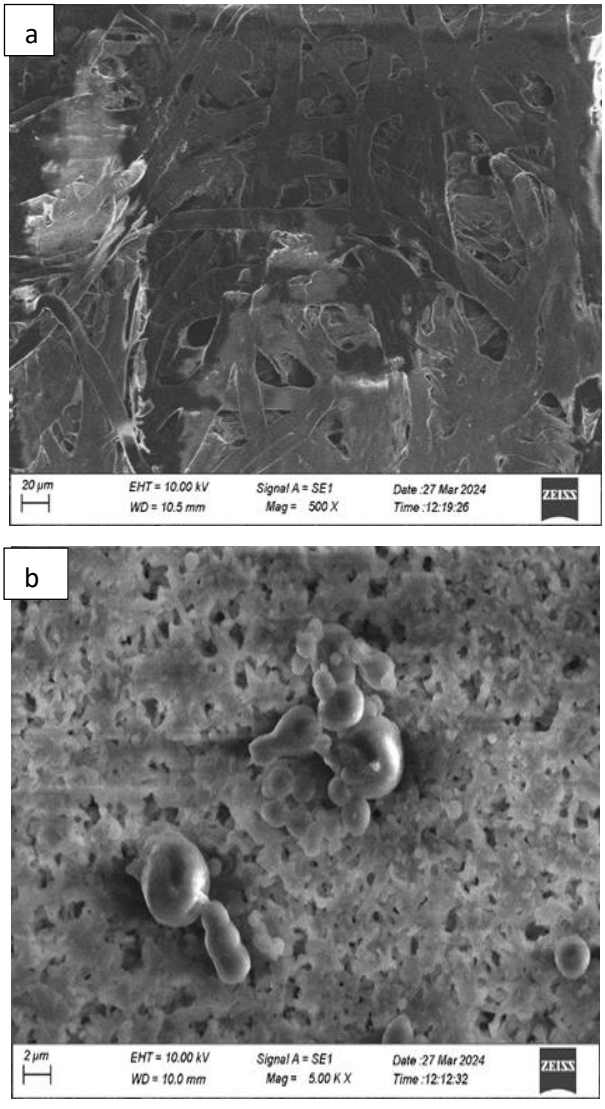


Fig 3a,3b,3c,3d- FTIR spectrum of blank and water samples

FTIR peaks obtained for blank, water samples are represented in Figure 3a,3b,3c and 3d. As Nylon membrane filter is used for filtering the microplastics, the following peaks were obtained for blank sample. Peaks at 3300,1277 cm^{-1} corresponds to N-H stretching [34]. C-H stretching of nylon is observed as peak at around 2934 and 2859 cm^{-1} [35] C=O stretching vibration of the amide group (-CONH-) is seen at 1632cm^{-1}[36] and it is a strong indicator of presence of nylon Peak at 1537 and 1538 cm^{-1} corresponds to N-H bending and C-N stretching [37] C-H bending around 1375–1450 cm^{-1},CH_2 bending at 1199 cm^{-1}, NH bending and C=O bending at 688cm^{-1}.

Like the blank, the respective peaks were obtained in all the water samples. In sample 2 a peak at 2363 cm^{-1} in an FTIR spectrum is often associated with the presence of carbon dioxide (CO_2) or carbonate species. This absorption typically corresponds to the C=O stretching vibration in CO_2 or bicarbonate (HCO_3^-) and carbonate ions [38].

In all the water samples an addition peak is obtained at 533 cm^{-1}. The presence of peak may be due to presence of mineral salts like calcium carbonate ($CaCO_3$) or magnesium carbonate ($MgCO_3$) [39].



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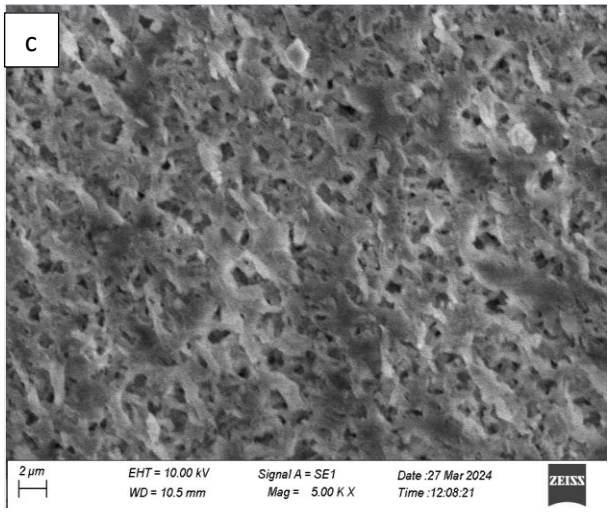


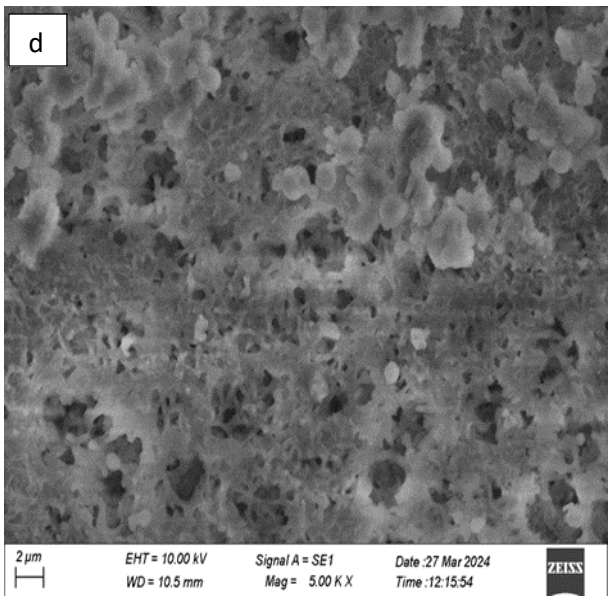
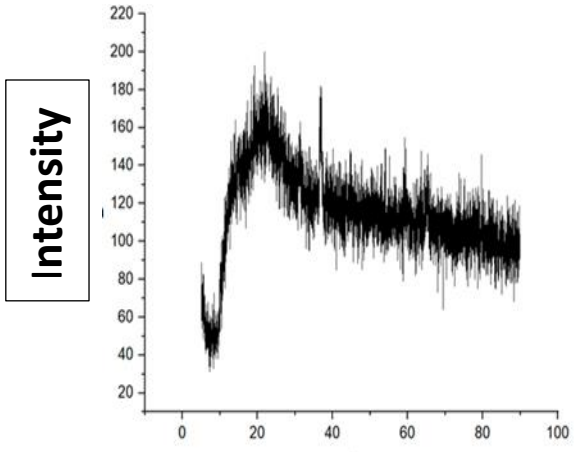
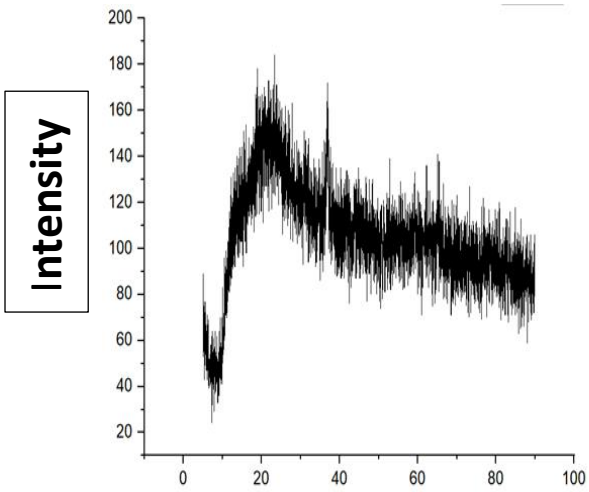
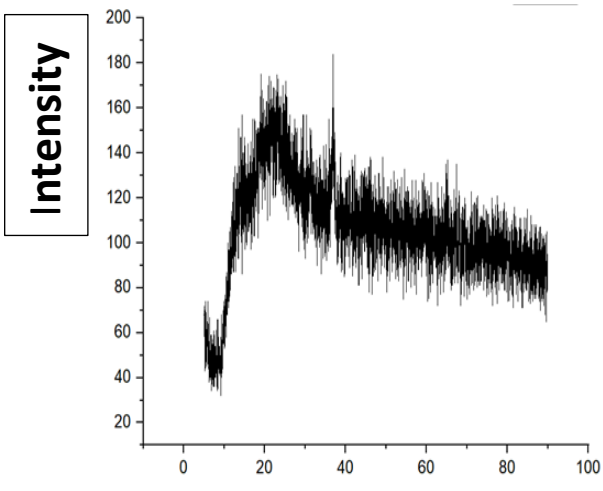


FIG 4a,4b,4c,4d SEM analysis of blank and water samples under 500x magnification

SEM is a useful tool for identifying MPs because it may produce highly magnified, clear images of microplastic particles that help distinguish them from organic particles. The Scanning Electron Microscope (SEM) analysis was performed to characterize the surface composition of the microplastic particles in order to identify their morphology [5]. In present study we analysis four samples were anayzed each with 2µm size in 500x magnification. The SEM image of water samples reveals that there is no agglomeration in the surface. SEM image 4c shows distinct small sized particles which may be microplastics.







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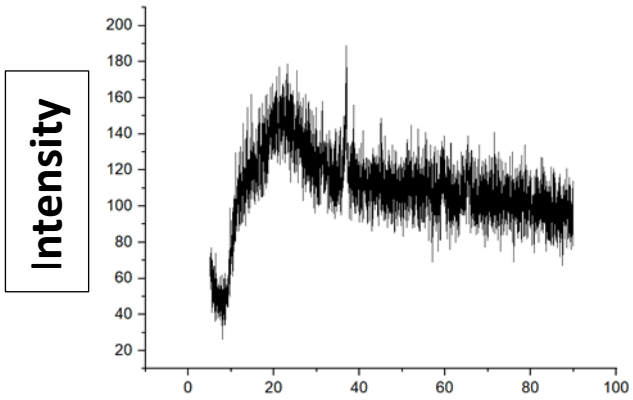

Fig 5a,5b,5c,5d XRD chromatogram

Fig 5a,5b,5c,5d represents the XRD chromatogram analyzed using ORIGIN software.broad peaks obtained in the images indicate the presence of amorphous or partially crystalline material which may be microplastics.

CONCLUSION

The physical and chemical characteristics of bottled and tap water samples were examined and all water samples complied with BIS standards for colour, pH, fluoride, and nitrate, confirming their safety for drinking. Tap water showed elevated levels of nitrite, nitrate, and ammonia, indicating possible pollution from organic sources or sewage. Microplastics were found in the sample, with distinct FTIR peaks showing their makeup, such as nylon and carbonate compounds. Extra peaks observed at 533 cm^{-1} in FTIR spectra indicate the presence of mineral salts like calcium or magnesium carbonates. SEM analysis showed microplastics of small size with unique shapes, while XRD data verified that they were either amorphous or partially crystalline in structure. Further advanced analytical techniques can be applied to confirm the presence of microplastics in the water samples.

Conflict of interest: NIL

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