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QUANTIFICATION OF MICROPLASTIC BY FT-IR AND ICP-MS

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ABSTRACT

Microplastics (MPs) are characterized as particles which are smaller than 5 mm. Microplastics (MP) pose significant threats to ecosystems, human health, and biodiversity by accumulating in food chains, disrupting organisms physiology, and potentially causing long-term environmental harm. This study investigates the prevalence of MPs at two major landfill locations in Chattogram, Bangladesh. A total of 30 samples were collected from soil during the dry seasons. The samples preparation involves sieving, chemical digestion, and density separation methods. Chemical identification and polymer detection was studied using Fourier transform infrared (FTIR) spectroscopy. ICP-MS used to quantify the MPs indirectly. Zn metal used to dope with MPs polymer PET and PP. This polymer later digested in microwave and hence the percentage doped in MPs was computed. The unbounded Zn was also analyzed in ICP-MS. This technique helps to indirectly quantify the MPs. This study underlined that landfill soils contains numerous MPs polymer such as polypropylene (PP), polyethylene terephthalate (PET), polystyrene (PS), low-density polyethylene (LDPE), and polyvinyl chloride (PVC) being the most common. The ICP-MS signifies as an alternative tool to quantify MPs in mass level that exacerbates the limits of particle counting method. This work in helps to understand the fate and occurenece of MPs in landfill leachate and contribute to sustainable waste management.

***Keywords:** FT-IR,, ICP-MS, , Metal doped MPs*

1. INTRODUCTION

Plastics have become an integral part of our daily activities which stored 335 million tons annually due to its portability, availability, and low cost. Despite growing awareness of plastic pollution and efforts to reduce usage, consumption continues to rise, further increased by the COVID-19 pandemic through increased demand for plastic-based personal protective equipment such as masks and gloves. Environmental degradation of plastics occurs by physical, chemical, and biological degradation processes and hence breaking down into smaller form known as microplastics (MPs, <5 mm). The environmental concern is spreading widely due to the use of synthetic fibers such as polyester, polyamide, acrylic, and polyolefin. Majority of these fibers contributes to 60% of global fiber consumption and are used in textiles for clothing, upholstery, agriculture, and medical applications, and they continuously shed fibers during wearing and laundering, releasing large quantities of synthetic microplastic fibers into domestic wastewater. Microplastic severly sustain in the enovironmnetal system which later migrate to aquatic sources and pose significant risk to the ecosysetem. Serious health risk such as potentially causing oxidative stress, tissue damage, inflammation, and the release of toxic compounds (Naveen et al., 2018; Parvin et al., 2021; Kabir et al., 2023)

Landfills are major sources of microplastic release by means of leachate that carries plastic fragments, fibers, and numerous contaminants into nearby soil and groundwater(Aziz et al., 2013; Parvin et al., 2021). In countries like Bangladesh, non-engineered landfill sites often lack adequate leachate collection or treatment systems which subsequently allows microplastics and heavy metals to migrate into nearby ecosystems(Kabir et al., 2023). Although microplastic pollution has received global concern and some studies have addressed their quantification and characterization in landfill leachate, where organic and inorganic matter complicate analysis. Conventional methods such as ATN-FTIR and micro-FTIR spectroscopy only characterize the chemical composition of microplastics in such complex matrices. However, most of the study measures the concentration by particle countinge which always not reliable for complex matixes. Therefore, advanced approaches like indirect quantification using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) are imperative to improve detection sensitivity, precisely assess microplastic concentrations and associated environmental risks in landfill and soil systems .

Fourier Transform Infrared (FTIR) spectroscopy is one of the most widely used analytical techniques for identifying and characterizing microplastics (MPs) in complex environmental matrices such as landfill leachate, sediment, and wastewater. A unique spectral fingerprint can be matched to known polymer libraries by which the frequency of inter molecular bond can detect the polymer in FTIR (He et al., 2019). Numerous polymer libraries such as polyethylene (PE), polypropylene (PP), polystyrene (PS), and polyethylene terephthalate (PET) which are the most frequently prevalent plastics in landfill leachates and other contaminated media are patent in the libraries (Kazour et al., 2019). The usability and non-destructive nature of FTIR make it a preferred technique for polymer identification across a wide range of particle sizes and environmental matrices.

For accurate detection in FT-IR sample preparation is challenging. In environmental samples such as leachate and sludge often contain high levels of organic matter, salts, and mineral residues. Firstly, samples are dried (50–60°C) and oxidative digestion using hydrogen peroxide (H_2O_2) of 20 mL in addition to Fenton's reagent to remove organic material(Rafiq et al., 2023). The cleaned residue is then processed through density separation using high-density saline solutions such as zinc chloride ($ZnCl_2$; 1.6–1.8 g/cm^3) to isolate plastic particles from heavier sediments. The recovered floating fraction is filtered through cellulose nitrate membranes (0.45–5 µm), rinsed with distilled water or ethanol to remove residual salts, and oven-dried before FTIR measurement (Hossain et al., 2021)

Different FTIR modes are used depending on particle size and sample characteristics. The Attenuated Total Reflectance (ATR-FTIR) mode is commonly used for MPs larger than 1 mm, while Micro-FTIR (µ-FTIR) is suitable for particles smaller than 1 mm and can detect MPs as small as 10–20 µm . However, FTIR analysis faces challenges when dealing with transparent, colored, or weathered microplastics and surface degradation or pigment interference may obscure characteristic peaks (Hidalgo-Ruz et al., 2012).Recent advancements, such as FTIR imaging and automated mapping systems and spatial resolution which allows for more accurate polymer identification in heterogeneous matrices (Rahmani et al., 2023). Overall, FTIR is reliable a technique for chemical characterization of microplastics, providing promising tool in polymer types detection.

Indirect determination of microplastics (MPs) using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) has recently gained attention as a reliable and quantitative alternative to traditional identification methods(Keller et al., 2020). Conventional microscopic and spectroscopic techniques, such as Fourier Transform Infrared (FTIR) and Raman spectroscopy, are widely used for microplastic characterization but often face significant limitations in detecting smaller particles, especially in complex environmental samples like sludge or soil. To overcome these analytical challenges, researchers have developed metal-doped or tracer-labeled microplastics that allow indirect quantification by measuring the associated metal concentrations using ICP-MS (Keller et al., 2020; Tran et al., 2024)

In this approach, trace metals such as indium (In) or zinc (Zn) are uniformly embedded within polymer matrices, enabling the use of metal content as a conservative tracer to indirectly estimate microplastic concentrations(Hildebrandt et al., 2020; Redondo-Hasselerharm et al., 2021; Tran et al., 2024). For example, specially designed indium-doped synthetic fibers have been used to quantify microplastic fibers and microwave digestion is a prerequisite for ICP-MS. The In doped with MPs is dissolved and later on the indium concentration measured via ICP-MS is correlated with fiber mass(Hildebrandt et al., 2020; Redondo-Hasselerharm et al., 2021; Tran et al., 2024). This method increased detection sensitivity and precision, particularly for microplastics in complex matrices where optical identification becomes difficult (Samrat Hossain et al., 2022). Moreover, the stable incorporation of metals within polymer structures ensures minimal leaching, allowing precise and reproducible quantification even at trace levels (Lin et al., 2023).

Recent studies have demonstrated the potential of ICP-MS-based indirect quantification in assessing the fate and transport of microplastics in environmental systems such as wastewater treatment plants and sludge amended soils. By doping metal in MPs intensity with the plastic mass balance, this approach also enhances accurate tracking of microplastic behavior, retention, and mobility across different environmental compartments. Therefore, the ICP-MS-based indirect quantification method represents a promising and powerful complementary tool to conventional spectroscopic techniques.

Microplastic characterization and quantification studies often limited to FT-IR technique which may exhibit false positive results in some context. On the contrary, ICP-MS indirectly quantifies MPs if it is blended properly with metal. This study brings the novelty of using ICP-MS of microplastic studies.

The main objective of thus study is to characterize the plastic polymer that are often disburse in the landfill and landfill leachate. FT-IR technique is adopted in this study for the characterization of microplastic. In addition, Inductively Coupled Plasma Mass Spectrometry is used for the the indirect quantification of MPs. Metal doping study in MPs and their retention in MPs was investigated in this study which subsequently help to understand the fate of MPs in landfill leachate. This integrated analytical framework provides a reliable and efficient strategy for advancing microplastic research in diverse environmental systems.

2. METHODOLOGY

2.1 Study Area

The metro area of Chittagong is around 2,510 km², whereas the urban area is 2,054.90 km². The total area of the metro is 5,282.92 km². 19,800 km² is the average population density there (Aziz et al., 2021). Currently, CCC dumps wastes into Ananda Bazar, Halishahar, S1 (22°21′5.02″ N and 91°45′6.07″ E) and Arefin Nagar, S2 (22°23'14.2"N and 91°47'51.1"E) open dumping sites. S1 and S2 both sites were choosen as study area. Coordinates of the sampling point were recorded (Table 1), and maps of the sampling point are shown in Figure 1 (a) and (b). For microplastic analysis, the samples were collected from the first week of April 2024 to the last week of May 2024 in the dry seasons. Four samples; Groundwater (GW), surface water (SW), leachate (L), and soil (S) were collected from three different locations at each dumping site.

Figure 1:Map of sampling points, (a) Ananda Bazar dumping station (S1), (b) Arefin Nagar dumping station (S2). Source: Google earth

2.2 Sample Collection

2.2.1 Soil Sample

Using a stainless-steel sampler, roughly 0.5 kg of soil was taken at each sampling location, ranging in depth from 0 to 30 cm. Before the analysis, three samples were taken from each location and thoroughly combined in a clean plastic container to create a composite sample. The soil samples were taken and brought to the lab. After that, they were ground into a fine powder using an acid-washed commercial mortar and pestle and sieved through a 0.425 mm mesh sieve (Wijeyaratne et al., 2021). It was then stored in labeled polythene bags. Soil sample collection site is shown in table 1.

Table 1:Coordinates of sampling points

Ananda Bazar dumping station		**Arefin Nagar dumping station**	
Points	Coordinates	Points	Coordinates
P1	Latitude: 22°18'42.57"N Longitude: 91°46'22.39"E	**P4**	Latitude: 22°23'14.2"N Longitude: 91°47'51.1"E
P2	Latitude: 22°18'46.5"N Longitude: 91°46'14.3"E	**P5**	Latitude: 22°23'15.07"N Longitude: 91°47'52.80"E
P3	Latitude: 22°18'47.41"N Longitude: 91°46'26.79"E	**P6**	Latitude: 22°23'12.81"N Longitude: 91°47'52.34"E

2.2.2 Ground Water, Surface Water and Leachate

In accordance with conventional protocols, water from the surface area was sampled 10-15 cm below the surface using marked acid-washed plastic containers to prevent unforeseen changes in characteristics and acquire a 1000 ml sample for surface water, groundwater, and leachate analysis (Reza and Singh, 2010). Each label for the water included information on the place and source from where it was sourced. To protect the metals and prevent precipitation, 2 milliliters of analytical-grade nitric acid were added to the water to acidify it (Kar et al., 2008). The water samples were kept cold, at 5ºC, until they were taken to the lab for examination.

2.3 Density Separation and Digestion

2.3.1 Processing for Soil Sample

The soil sample were oven-dried at 60°C for 1 hour, sieved through a 75 µm sieve, and taken as 5 gm. For density separation, 100 ml of $ZnCl_2$ (1.8 gm/cm^3) is added and stirred well and kept for 24 hours. For oxidizing and separating the supernatant, 5 ml of 30% H_2O_2 and 5 ml of $FeSO_4$ (0.05 M) were added to remove organic matter. The samples were kept in a water bath for 1 hour for digestion and the solution was cooled several times. During the oxidizing process, subsequently 30% more H_2O_2 can be added to have a clear solution. The supernatant was separated again and filtered through a 0.45 µm cellulose membrane filter through a vacuum pump. After filtering, the filter paper was kept in a petri dish at room temperature for 24 hours for further testing.

2.3.2 Processing for Groundwater and Leachate

The surface water and leachate samples were taken as 250 ml and sieved through a 200 µm sieve to remove the floating particles. For oxidizing, 5 ml of 30% H_2O_2 and 5 ml of $FeSO_4$ (0.05 M) were added to remove organic matter. The samples were kept in a water bath for 1 hour for digestion and the solution was cooled several times. During the oxidizing process, subsequently 30% more H_2O_2 can be added to have a clear solution. For density separation, the supernatant is separated, 5 ml of $ZnCl_2$ (1.8 gm/cm^3) is added and stirred well, and it is kept for 24 hours. The supernatant was separated again and filtered through a 0.45 µm cellulose membrane filter through a vacuum pump. After filtering, the filter paper was kept in a petri dish at room temperature for 24 hours for further testing.

2.4 Polymer Identification

The most used spectroscope for analyzing microplastic is the Fourier transform infrared (FTIR) and Raman spectroscope (Hidalgo-Ruz et al., 2012). First, the microplastics that shared the same appearance were taken out of the filter papers and put into a little glass vial. After that, the glass vial was examined to identify the type of polymer. A Perkin Elmer Fourier Transformed Infrared Spectroscopy (IR spectrum ES version 10.6.2) was used to conduct the tests. Utilizing the Attenuated Total Reflection (ATR) technique, the samples were measured. FTIR-ATR can be used to determine whether functional groups and bonds are present in the fiber polymers or not. The wavelengths were changed from 4000 to 400 cm^{-1}. spectrum of each plastic polymer that had been disclosed in previously published literature. In this study, the sample was prepared at Chittagong University of Engineering and Technology (CUET), and microscopic and FTIR tests were done at the Wazed Miah Science Research Center, Dhaka University.

2.5 Sample Preparation for ICP-MS

2.5.1 Microplastic Doping with Metal

To quantify microplastics indirectly, zinc (Zn) metal was employed as a tracer element. A 5 ppm zinc chloride ($ZnCl_2$) solution was freshly prepared using analytical-grade reagents and deionized water. The prepared $ZnCl_2$ solution was used for the doping of PET and PP MPs separately. For the doping process, 100 mg of each microplastic sample was mixed with the $ZnCl_2$ solution. The mixture was subjected to mechanical stirring at a speed of 250–300 rpm for 5–6 hours to ensure proper interaction between Zn ions and the microplastic surfaces.

After stirring, the mixture was kept for 20–30 minutes to allow stabilization so that adsorption of Zn ions onto the MPs. The resulting suspension was filtered through Whatman Grade B(8 µm) filter paper. During filtration, the doped microplastic particles were retained on the filter surface amd the filtrate containing the unbound Zn ions was collected separately. The filtrate was taken in small aliquot for analysis by Inductively Coupled Plasma Mass Spectrometry (ICP–MS) to determine the residual zinc concentration. The whole process of MPs doping is shown in Figure 2.

2.5.2 Microwave Digestion of Doped Microplastic

The retained doped MPs on the filter paper were transferred cautiously and dried in an oven at 50 °C for 6–8 hours to remove any moisture content. The dried microplastic powders were used for digestion prior to ICP–MS analysis. The digestion process was conducted using a microwave digestion system. Approximately 100 mg of the doped MP sample was placed in a Teflon digestion tube. Five milliliters of deionized water were added to each tube to enhance the acid microwave digestion process.

The digestion was performed at a temperature of 250 °C and a pressure of 120 bar to ensure complete breakdown of the polymer matrix and release of the bound zinc ions are shown in table 2. After digestion, the solution was allowed to cool to room temperature. The digested mixture was then diluted to a final volume of 50 mL with Milli-Q water. A small aliquot of this diluted solution was taken for ICP–MS analysis.

Table 2: Microwave Digestion of Polymer

MPs Polymer	Weight(mg)	DI Vol(mL)	Temp(Celcius)	Pressure	Digestion Time(min)
PET	50	5	250	120	60
PET	100	10	250	120	60
PP	50	5	250	120	60

A. MPs Sieving(50-200 um) **B. MPs Sample** **C. Mechanical Stirring** **D. Filtration and Drying**

E. Microwave Digestion **F. Doped MPs Solution** **G. Teflon Tube Sample for ICP-MS** **H. ICP-MS Analysis**

Figure 2: Metal Doped Microplastic detection in ICP-MS

3. RESULTS

3.1 FT-IR Analysis

FTIR-ATR spectrums are explained in different samples below. Most of the plastics were found in leachate, surface water, and soil samples, respectively. Each type of polymer was shown for each sample according to their spectrum 67.71% of the examined samples were found to be known polymers. Seven different kinds of polymers were found in groundwater, with low-density polyethylene (LDPE) and polyvinyl chloride (PVC) predominating. At S1 (Ananda Bazar Dumping Site), LDPE accounted for 43.23% of the total, while at S2 (Arefin Nagar Dumping Site), PVC made up 36.59%. Six different types of polymers were found in surface water, with polyethylene (PE) and polyethylene terephthalate (PET) being the most common, accounting for 39% and 27% at S1 and 50% and 36% at S2, respectively. Ten different polymer types were found in leachate samples, which had the highest polymer diversity. Polypropylene (PP) predominated, accounting for 35.04% in S2 and 28.56% in S1. Similarly, out of the ten polymer types found in soil samples, LDPE was the most prevalent (45.08% in S2 and 28.08% in S1), followed by HDPE and PET. This suggests that LDPE was the predominant microplastic in the majority of sample types and locations. This are shown in (Figure 3).

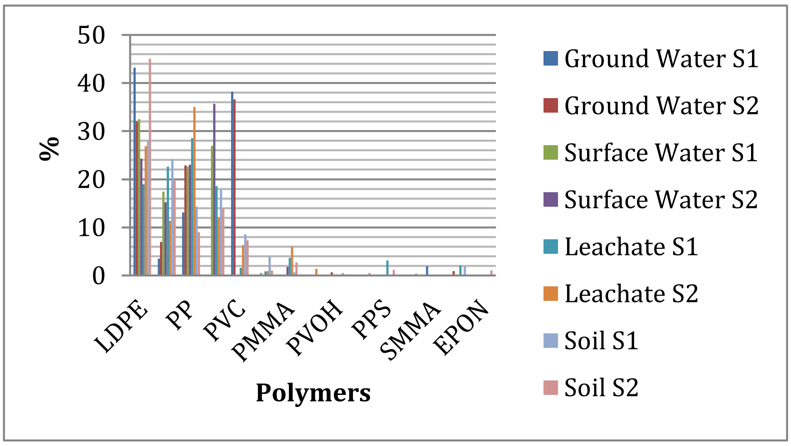

Figure 3:Different types of polymers identified (PP, SMMA, DPMPI, PVOH, PC, HDPE, LDPE, PVC, PMAA, PET, PPS, SMA, PEU, PTT, and EPON.) (S1: Ananda Bazar Dumping site, S2: Arefin Nagar Dumping site)

For the primary polymers (Figure 4), the FTIR-ATR analysis revealed distinct spectral characteristics. Isotactic polypropylene (PP), the most common industrial variant, exhibited a prominent CH_3 peak at 1375 cm^{-1} and clear mid-IR bands ranging from 1330 to 809 cm^{-1}, with additional bands appearing at 740–710 cm^{-1} after being copolymerized with ethylene. Polyethylene terephthalate (PET) has trans and gauche conformations; the bands were observed at 1466–847 cm^{-1} and 1449–896 cm^{-1} for C=O and CH_2, respectively. LDPE showed strong bands due to the presence of branching methyl groups at 1378 cm^{-1}, while HDPE exhibited weaker bands. The presence of crystallinity is indicated by sharp rocking modes near 720/730 cm^{-1} and sharp peaks at 1175 and 1050 cm^{-1}. Its presence was established through strong C–Cl stretching (700–550 cm^{-1}) and CH_2 bands (3000–2800 cm^{-1}) in the infrared spectrum of PVC with variations in spectra brought about by different plasticizers.

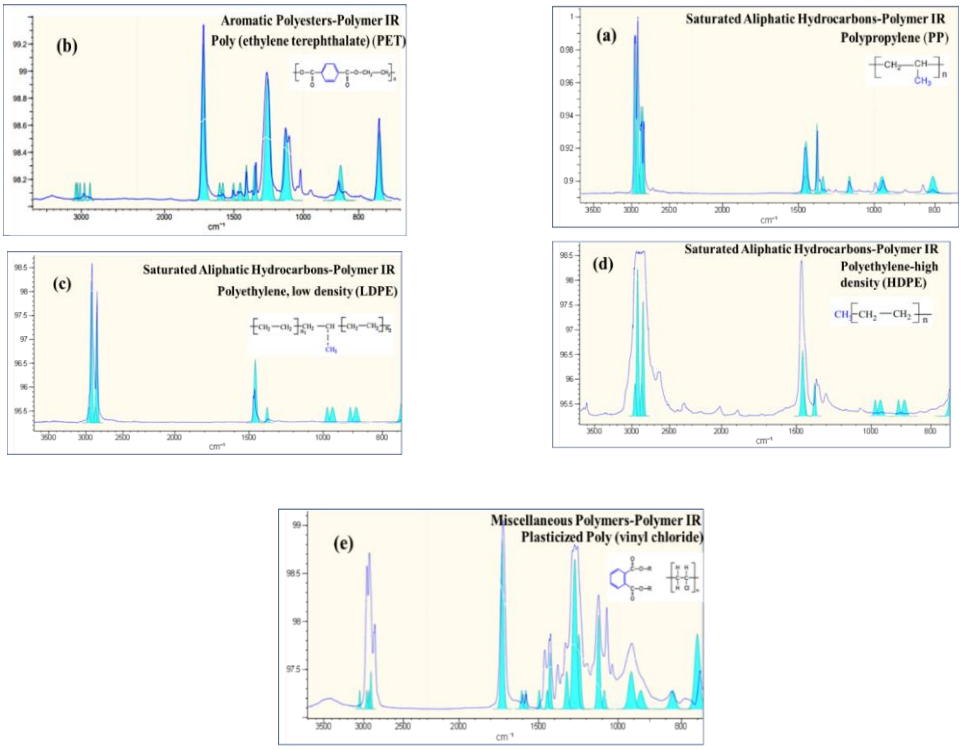

Figure 4:FTIR-ATR Spectrum (Transmission (%) vs. Wave Number (cm^{-1})) of; (a) polypropylene (PP); (b) Poly (ethylene terephthalate) (PET); (c) Polyethylene, low density (LDPE); (d) Polyethylene-high density (HDPE);(e) Plasticized Poly (vinyl chloride) (PVC)

3.2 ICP-MS Analysis

The quantification of zinc-doped microplastics (MPs) was conducted to ensure the efficacy of metal adsorption on polymer surfaces. This indirect method provides later on to understand the fate and interaction behavior of MPs with trace metals in aqueous environments. The results obtained from ICP–MS analyses shows satisfactory doping and recovery values for both PET and PP microplastics.

Microplastic sampling and Zn doping were found to be challenging but controlled study comp;ted satisfactorily. Zinc chloride ($ZnCl_2$) of 2.08 g was dissolved to prepare a 2000 ppm stock solution, which was subsequently diluted to obtain a 5 ppm working solution for doping. The accuracy of the Zn standard was validated through ICP–MS using internal standards of 0.1, 0.5, and 1 ppm, all of which produced reliable calibration results. The blank deionized water (DI) solution exhibited negligible Zn concentrations, confirming the absence of contamination.

A 5 ppm Zn solution which was taken for the confirmation of ICP-MS accurately reflected the expected intensity counts in ICP–MS. For the doping experiment, 100 mg each of PET and PP MPs were mixed separately with the Zn solution separately. After mechanical stirring and stabilization, Zn adsorption onto the microplastic surfaces was confirmed through subsequent microwave digestion and ICP–MS analysis. The digested PET MPs exhibited a Zn concentration of 2.316 ppm, indicating partial adsorption of Zn ions on the polymer surface. This are illustrated in table 3.

Table 2:Doped Microplastic Metal Concentration after Microwave Digestion

Metal	**Concentration(mg/l)**	**ICP-MS(mg/L)**
Zn(PET)	-----	2.316
Zn(PE)	-----	3.312
Zn	5	5.312
Blank DI	-----	0.10

The filtrate obtained after separation showed a Zn concentration of 3.369 ppm. The combined Zn value of 5.685 ppm slightly exceeded the initial concentration by about 10% which may be incorporated to analytical uncertainty or matrix effects during ICP–MS measurement. Similarly, for PP MPs the digested sample exhibited a Zn concentration of 3.312 ppm while the filtrate contained 2.895 ppm of Zn. The total Zn concentration of 6.207 ppm was approximately 20% higher than the initial 5 ppm solution that suggest possible aggregation or incomplete recovery during filtration. This result are shown in table 4.

Table 3:Metal Concentration of Zinc Chloride Solution after doping MPs

Metal	**Concentration(mg/l)**	**ICP-MS(mg/L)**
Zn	5	5
Zn	-----	3.369
Zn	-----	2.895
Blank DI	-----	0

Table 4: Microplastic Doped Percentage

Polymer	**Weight(mg)**	**Initial Metal Concentration(ppm)**	**Doped MetalConcentration(ppm)**	**Doping(%)**	**Indirect Mass Qunatification(ppm)**
PET	100	5	2.316	47%	47
PP	100	5	3.312	60%	60

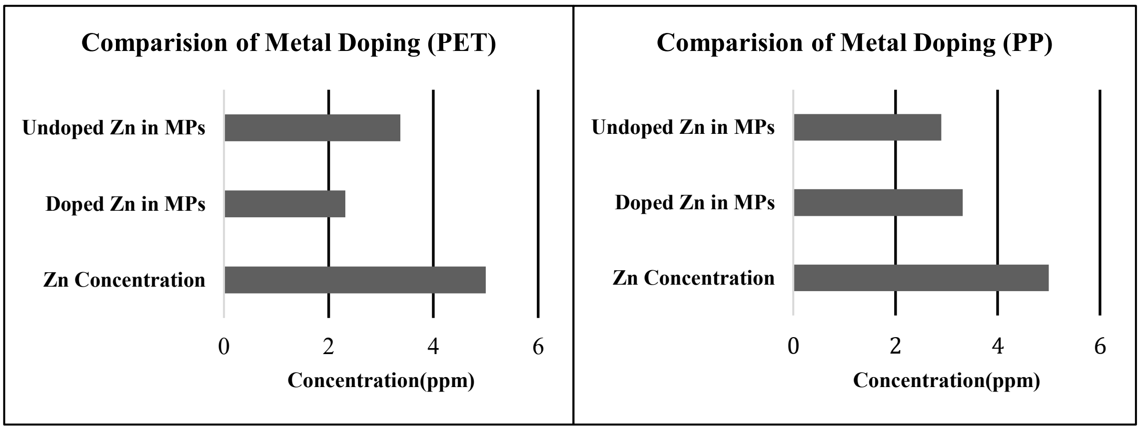

Figure 2:Metal Doping Percentage in PET and PP

4. CONCLUSIONS

In this study FT-IR was used to chemical characterization of microplastic polymer in two main landfill of Chattogram city. The Zn metal doping was done on microplastic to indirectly quantify the mass of MPs was also conducted in this study by ICP-MS. The major findings is given below:

- Identifiable polymers accounted for 67.71% of all recovered particle dominated by LDPE 45.08% in soils and 43.23% in groundwater(S1) and PVC reaching a maximum of 36.59% in S2 groundwater.
- Microplastic was highest in leachate in which PP was 35.04% that indicates strong plastic degradation and transport across the dumping site.
- The doping percentage of Zn metal on PET and PP was 47% and 60% which indicates PP is more detectable more adsorbing Zn on PP by ICP-MS shown in Figure 5.
- The undoped Zn metal after filtering was also analyzed separately which only excceds 5-15% of total concentration of Zn solution 5 ppm. This result indicates the quality control and precision of ICP-MS is accurate.

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Declaration of Use of AI:

Authors use AI in the introductory part of this paper only for sentence coherence.

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