

**Occurrence, Distribution and Composition of Microplastics in Bays and Estuaries of the  
Texas Coastal Bend**

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Prepared by:

Jordan Cisco, Xiangtao Jiang, Jessica Arisbeth Urias-Quiroz, Kaijun Lu, Jianhong Xue, and  
Zhanfei Liu

University of Texas Marine Science Institute

750 Channel View Dr. Port Aransas, TX 78373

(361) 695-4732

Email: zhanfei.liu@utexas.edu

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**Coastal Bend Bays & Estuaries Program**

615 N. Upper Broadway, Ste 1200 Corpus Christi, Texas 78401

## **Executive Summary**

Plastic pollution is becoming a severe environmental issue globally because of the increasing production of plastics and their resistance to environmental degradation, leading to their accumulation in natural waters, mostly in the forms of micro- and nanoplastics. While the bioaccumulation of MPs in marine biota has long been demonstrated, recent evidence has clearly shown that plastics can accumulate in human tissues or organs, presenting a direct threat to human health. Texas coasts are a relative hotspot for plastic pollution due to the abundance of petrochemical industries, including plastic manufacturers, dense populations, and the convergence of coastal currents that may facilitate the accumulation of plastic debris in bays and estuaries. Therefore, knowing the distribution and composition of microplastics in Texas bays and estuaries is of vital importance if we are to predict the seriousness of this environmental problem and implement appropriate policies for management. Expectedly, plastic pollution may endanger the health of coastal ecosystems with the continuous input of microplastics to bays and estuaries in the Coastal Bend, yet there is little data. In this project, we collected water samples from Copano Bay, Aransas Bay, Corpus Christi Bay, Nueces Bay, Upper Laguna Madre, and Baffin Bay for microplastic analysis. To do so, we first developed a streamlined protocol, including sample collection using plankton tow, chemical digestion using nitric acid, and quantification using pyrolysis gas chromatography mass spectrometry. The results showed that within Corpus Christi Bay, the concentration of microplastics was up to 1.6  $\mu\text{g/L}$  and the plastics were dominated by polyethylene (PE), polypropylene (PP) and polyethylene terephthalate (PET), together with styrene-butadiene rubber (SBR), nylon-66 (N66), and polymethyl methacrylate (PMMA) as the minor components. Using these results for a rough calculation, the whole Corpus Christi Bay may contain up to 1 metric ton of PE, as an example. This work offers the first direct quantification of microplastics of the bays and estuaries in Texas Coastal Bend. The results will help policy makers evaluate the source and degree of the plastics contamination and the potential ecological impact, and act appropriately to maintain healthy estuarine ecosystems.

## **1. Introduction**

Plastics are widely used in modern society due to their durability and versatility, but their persistence in the environment has led to widespread contamination, particularly in marine ecosystems. The global plastics production has increased over one thousand times from 0.3 to 348 million tons in 60 years (Plastic Europe, 2019) and will certainly continue to increase in the foreseeable future. Given the huge amount of production, however, only 40% of the plastic waste is recycled, with the rest ending up in landfills or eventually being washed out to oceans and lakes (Boucher and Friot, 2017; Geyer et al., 2017; Hendrickson et al., 2018). Plastic pollution in the marine environment is an emerging global environmental concern. It has been estimated that annually over 4.8-12.7 million tons of plastic waste end up in the ocean from land-based sources (Jambeck et al., 2015). In addition, plastics account for about 90% of marine debris (Wessel et al., 2019). With durable properties, plastic debris lasts for a long time, often years to decades, in

the marine environment, but they do undergo environmental processes such as weathering and fragmentation, producing numerous smaller-sized plastic debris (Mattsson et al. 2015).

Microplastics (MPs), defined as plastic particles smaller than 5 mm (Arthur et al., 2009), are of a growing concern due to their ability to accumulate in water bodies, sediments, and organisms. Microplastics in the marine environment could be further categorized into two groups based on their source: (1) primary microplastics originated from sources for direct use, including nurdles, virgin resin pellets, cosmetics, scrubs and abrasives (da Costa et al. 2016; Hernandez et al. 2017), and (2) secondary microplastics formed from the breakdown of larger plastics materials by mechanical forces in use or in the environment such as weathering or photooxidation (Mattsson et al. 2015). It is estimated that about 75% -90% of the plastics in the marine environment come from secondary products (Andrady, 2011). The presence of microplastics has been documented across all marine environments, from surface waters and sediments to deep-sea trenches and Arctic ice (Sul et al., 2013; Fang et al., 2018; Peng et al., 2020).

Due to their small size, MPs are ingestible to marine organisms such as crabs, oysters and fish (Crooks et al., 2019; Lu et al., 2016). It has also been shown that microplastics are transferable across different trophic levels through the food web (Crooks et al., 2019). Humans also suffer a risk of microplastic ingestion via microplastic-contaminated seafood or sea salts (Rochman et al., 2015; Li et al., 2015). Recently, there has been clear evidence of micro- and nanoplastics accumulation in human tissues that may cause health related issues (Marfella et al., 2024; Nihart et al., 2025). In addition, during the plastics production process, additives that have a wide range of toxicity such as plasticizers, antioxidants, ultraviolet stabilizers and flame retardants are often added to modify the properties of plastic products. Along with the additives, unreacted monomers, catalysts and by-products could all be present in plastics and could be desorbed or leached out once microplastics are in aquatic environments. Moreover, the hydrophobic nature and high specific surface area of microplastics make them great adsorbents for hydrophobic organic pollutants such as polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbon (PAHs) in environments (Jiang et al. 2019; Koelmans et al. 2009; Liu et al. 2018). High concentrations of organic pollutants have been detected from field microplastics. For instance, PAHs at concentrations of 0.014–0.85  $\mu\text{g/g}$  were measured on plastic debris collected from the North Pacific subtropical gyre (Chen et al. 2018). Once these contaminated microplastics are ingested by organisms, they can act as a vector for the exposure of pollutants to the organisms. Thus, it is of great importance to evaluate the toxic effects and ecological risks of microplastics in the marine environment. Such evaluation relies on knowing the spatial and temporal distributions of microplastics. However, the existing microplastic data is far from enough to systematically assess microplastic distribution in the marine environment. Studies on microplastic abundance generally focus on one-site sampling for one time, thus seasonal and long-term monitoring of microplastic abundance studies are rare.

Debris along the Texas coast has long been paid attention to. Since the 1990s, for example, it was found that the National Seashore in the north-western Gulf of Mexico (GOM) (in Texas) contained over 30 times more plastic debris than the other coasts along GOM (Miller and Echols, 1996; Ribic et al., 2011). In northern GOM, Wessel et al. (2016) found that microplastic concentrations were 66-253 times higher than microplastic concentrations in the open ocean, and the concentration is related to the presence and pattern of currents and tides. In addition, a recent survey found that plastic debris accounted for over 90% of all marine debris in GOM, and beaches of the Mission-Aransas Reserve accumulated 10 times more than other beaches of GOM (Wessel et al., 2019). Moreover, based on a citizen science project, “Nurdle Patrol”, large quantities of microplastic pellets (nurdles) were found along Texas beaches (Tunnell et al., 2020). Further work showed that PAHs, PCB and mercury have all been detectable on nurdles collected along the Texas coasts (Jiang et al., 2021; 2022). In addition, many petrochemical companies and plastic manufacturers have been established in this region, such as Formosa at Point Comfort and Exxon at Portland, Corpus Christi, which can potentially release plastics through wastewater discharge. Thus, the Texas coast including Coastal Bend suffers a high risk of microplastic exposure.

Given the widespread occurrence and potential ecological impacts of MPs in Texas bays and estuaries, accurate quantification, characterization, and monitoring are essential to understanding their environmental distribution and informing mitigation strategies.

### **1.1 Sampling and Analytical Protocol**

MPs in the marine environment have a wide range of size, shape, and polymer types, which make the analysis extremely challenging. For example, Fourier transform infrared (FTIR) and Raman spectroscopy are the go-to tools for polymer identification, but they only analyze plastics larger than 10s of microns and have requirements for certain morphology etc., such as flat surface when using microscopy FTIR. In contrast, microplastics in seawater are often in submicrons and as fibers (Koelmans et al, 2019). In addition, the amount of microplastics is dwarfed by the super high background of natural organic matter in the forms of organisms and detritus. The concentrations of microplastics in the ocean range from 0.002 to 62.50 pieces/ $m^3$  (Mutuku et al., 2024), whereas the amount of natural suspended particles should be easily in millions to billions with diameters of 10s-100s microns. For example, there are 100-20,000 diatom cells in only 50 ml of coastal seawater. Therefore, plastics need to be concentrated and isolated from natural organic matter before chemical analysis, which otherwise would be impossible to analyze. As such, researchers have developed a wide range of techniques for sampling, processing, and analyzing plastics in the marine environment (Stock et al., 2019). However, existing methods vary widely, leading to inconsistencies in data collection and interpretation. There is a critical need to develop a streamline protocol to collect, process and analyze microplastic samples from marine waters.

## *Field Sampling Techniques*

Accurate quantification of MPs in marine environments requires rigorous and standardized sampling methodologies to ensure comparability across studies. The two most employed techniques for MP collection from surface waters are plankton net tow sampling and bulk water sampling. Each method differs in operational approach, size-selectivity, and potential biases, necessitating careful consideration of their respective applications.

Plankton net tow sampling is one of the most widely utilized methods for MP collection, particularly for surface waters. This technique involves towing a neuston or plankton net, typically with a mesh size ranging between 100–500  $\mu\text{m}$ , at a controlled speed and duration, allowing the capture of floating MPs within a defined size range (Lindeque et al., 2020). The cod-end collection system ensures the retention of MPs and other particulate matter for subsequent processing. One of the primary advantages of this method is its ability to sample large volumes of water over a broad spatial scale, making it particularly suitable for assessing spatial distributions of MPs in marine ecosystems. Additionally, standardizing net dimensions and towing speeds enhances reproducibility and facilitates cross-study comparisons.

However, plankton tow sampling exhibits size-dependent selectivity, primarily capturing MPs larger than the net's mesh size while potentially underestimating smaller MPs (<100  $\mu\text{m}$ ) (Di Mauro et al., 2017). Studies have shown that MPs retained in neuston, or manta trawl nets may only represent a fraction of the total MP burden, as smaller particles pass through the mesh (Tikhonova et al., 2024). Furthermore, environmental factors such as surface currents, wave action, and turbulence influence MP retention efficiency, potentially introducing variability in particle concentrations across replicate tows.

In contrast, bulk water sampling involves collecting a fixed volume of water using glass or metal containers, which is then filtered in the laboratory to isolate MPs (Hung et al., 2020). This approach allows for the detection of a broader range of MP sizes, including particles below 100  $\mu\text{m}$  that would otherwise be excluded in net-based methods. Bulk water sampling is particularly advantageous in environments where MP concentrations are low, as it enables the concentration of MPs from large water volumes through high-efficiency filtration systems.

Despite its advantages, bulk water sampling is inherently limited by logistical constraints, as processing large water volumes requires substantial filtration time and capacity (Razeghi et al., 2021). Additionally, sample heterogeneity can introduce high variability, particularly in regions with high MP patchiness, where small-volume grabs may not be representative of broader distributions. Studies comparing plankton net tows and bulk water collection have reported up to fourfold differences in MP concentrations, highlighting the impact of sampling methodology on reported MP abundances (Barrows et al., 2017).

A combined methodological approach—integrating plankton tow sampling for broad-scale assessments with bulk water sampling for high-resolution particle size analysis—is increasingly recommended to obtain a more comprehensive representation of MP contamination in surface waters (Liu et al., 2020). Standardization of tow speed, mesh size selection, and water volume filtration remains a key priority for improving data comparability across studies and ensuring robust environmental monitoring.

This study adopted plankton tow sampling due to its advantages in large-volume collection, spatial consistency, and methodological reproducibility, ensuring a comprehensive assessment of MP distributions in the Texas Coastal Bend bays and estuaries.

### *Sample Processing and Treatment*

The selection of an appropriate chemical digestion method is critical in MP research, as it directly influences MP recovery, integrity, and the accuracy of subsequent polymer characterization. The primary objective of digestion is to remove natural organic material while preserving the physical and chemical structure of MPs, ensuring reliable identification and quantification. However, various digestion protocols—oxidative, alkaline, and acidic—demonstrate different degrees of efficiency, selectivity, and polymer stability, necessitating a careful balance between organic matter removal and plastic retention.

Oxidative digestion, typically using hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 30–35%), has been widely implemented due to its ability to effectively degrade organic material while maintaining high plastic recoveries. Studies indicate that short-term exposure (e.g.,  $\leq 48$  hours) at room temperature yields recovery rates exceeding 95% for most polymer types, including polyethylene (PE), polypropylene (PP), and polystyrene (PS) (Tuuri et al., 2024). However, prolonged exposure (e.g., 7 days) can induce morphological changes in certain polymers, particularly PP, despite maintaining high mass recoveries ( $\sim 97.5\%$ ) (Nuelle et al., 2014). The reaction kinetics of oxidative digestion can also vary depending on organic matter composition, necessitating process optimization to maximize the removal of natural organic matter, but without polymer degradation. Sodium hypochlorite ( $\text{NaClO}$ ) is sometimes used as an alternative oxidative reagent, but its potential to modify or destroy polymer structures, particularly polyamides, limits its application in MP analysis.

Alkaline digestion methods, primarily involving sodium hydroxide ( $\text{NaOH}$ ) or potassium hydroxide ( $\text{KOH}$ ), are commonly applied for biological matrices due to their effectiveness in protein hydrolysis. However, polymer stability varies significantly under alkaline conditions. Polyethylene terephthalate (PET) is particularly susceptible to degradation in highly alkaline environments, with mass recoveries dropping to 70–75% when subjected to 10 M  $\text{NaOH}$  (Olsen et al., 2020; Hurley et al., 2018). Reducing the concentration to 1 M  $\text{NaOH}$  improves PET retention, suggesting that controlled alkalinity is essential for maximizing polymer stability while

achieving effective digestion. Alkaline treatments are also known to induce swelling in polyamide (PA) and polyurethane (PUR), potentially affecting polymer integrity and spectroscopic identification. Despite these challenges, NaOH and KOH remain widely used for digesting biotic samples, as they offer efficient organic matter removal while being compatible with the most commonly found environmental MP polymers.

Acidic digestion methods vary significantly in their impact on MP integrity. Strong acid treatments, such as 22.5 M nitric acid (HNO<sub>3</sub>), have been reported to cause extensive polymer degradation, particularly in low-density plastics. Avio et al. (2015) demonstrated that only 4% of PE and PS remained intact following exposure to 22.5 M HNO<sub>3</sub> for 12 hours at room temperature, followed by 30 minutes of boiling. However, milder acid treatments appear to mitigate polymer loss. Schrank et al. (2022) reported that PE exhibited stability (95–100% recovery) when subjected to boiling 15.7 M HNO<sub>3</sub> for 2 hours, with PS recovery increasing to approximately 49%, suggesting that reduced acid concentrations and controlled heating can improve polymer stability while maintaining effective digestion.

It is important to optimize digestion conditions to retain a broad range of polymer types. A systematic review by Tuuri et al. (2024) evaluated different digestion strategies and found no universal advantage of oxidative versus alkaline digestion in terms of MP stability. Instead, recovery efficiency is highly dependent on reaction conditions, with lower reagent concentrations and shorter digestion times generally yielding higher plastic recoveries while still achieving sufficient organic matter removal. This highlights the necessity for tailored digestion protocols that consider sample composition, polymer stability, and analytical objectives, i.e., driven by specific samples from the environment.

The optimized digestion method developed in this study seeks to balance reagent concentration and reaction duration to achieve high MP retention across various polymer types while ensuring efficient organic matter degradation. By refining digestion parameters, we aim to establish a standardized approach that maximizes analytical reproducibility and minimizes polymer alteration, thereby enhancing the reliability of the MP results.

### *Analytical Techniques for Microplastic Identification*

The common instruments used to identify and quantify MPs include microscopy, FTIR, Raman spectroscopy, pyrolysis gas chromatography mass spectrometry (Py-GC/MS), thermogravimetry GC/MS etc. Microscopy techniques are straightforward but suffer large uncertainty because of the high similarity of natural particles to MPs in terms of morphology. While FTIR and Raman techniques offer quick identification of the polymer type, a plastic sample has to be in a size range of 10s of microns. For a thorough quantification, plastics have to be measured individually, which presents a tremendous challenge in terms of time and effort, or often unrealistic, because of the numerous plastic pieces on a chemically digested sample, often on filters. In addition, these techniques do not directly offer the amount of plastics in a given

sample, so to do so, some assumptions or computing have to be involved, such as the estimate of volume for a given piece of plastic and its density etc., which would lead to large uncertainty when extrapolating the whole sample and further a waterbody. In contrast, Py-GC/MS is based on mass spectrometry, therefore it is extremely sensitive in identifying and quantifying compounds in trace levels. With calibration standards, this technique directly quantifies the plastic sample, and because of the distinct characteristic ions for each polymer, it can analyze a sample consisting of many different types of plastics in one single run. As such, Py-GC/MS is a technique we chose for this project.

## **1.2 Project Goals**

In this project, we aim to refine and standardize protocols for sampling, processing, and analyzing MPs in marine waters, with a focus on bays and estuaries in the Texas Coastal Bend, where MP contamination is influenced by industrial and urban sources (Figure 1). By implementing a robust methodological framework, this study will provide reproducible baseline data to assess spatial variations in MP pollution, supporting future environmental monitoring and policy initiatives.

## **2. Materials and Methods**

### **2.1 Sampling**

The field sampling program was designed to collect microplastics samples in bays and estuaries of Coastal Bend. Sampling was conducted at 18 stations in Mesquite, Copano, Aransas bays, Corpus Christi and Nueces bays, Upper Laguna Madre and Baffin Bay (Figure 2). In July and October 2024, samples were collected from surface waters at each sampling station by tying a plankton net (150  $\mu\text{m}$  mesh, 50 cm diameter) at a speed of 2 knots allowing tidal current to flow through for approximately 10 min (Almeda et al., 2016). During the sampling, a high abundance of jellyfish in the area led to difficulty in sample collection, as they clogged the plankton net and cylinder. The jellyfish tissue also presented problems in the later stage of sample digestion. Therefore, we successfully designed and installed a jellyfish excluder, made of stainless-steel mesh with 2.2mm x 2.2 mm holes, on the top of the plankton net opening (Figure 3). The excluder can be easily attached or detached depending on the presence of jellyfish.

Contents of the collection buckets (cod ends) were filtered to separate microplastics. Other water and environmental quality parameters, such as specific conductivity, pH, and dissolved oxygen (DO) were also measured using a YSI Sonde (Table 3). Blank controls will be accomplished through filtering high purity water using the same procedure.

### **2.2 Digestion**



The digestion procedure was adapted from Tuttle and Stubbins (2023) with modifications to accommodate the high organic content present in the plankton tow samples. The optimized digestion method was designed to efficiently degrade organic material while preserving MP integrity for subsequent analysis. The digestion solution was composed of 70% nitric acid ( $\text{HNO}_3$ ) and sodium persulfate ( $\text{Na}_2\text{S}_2\text{O}_8$ ), ensuring effective oxidative degradation of organic constituents while maintaining polymer stability. Due to the high plankton biomass in the samples, direct filtration was unsuccessful; therefore, sample-specific digestion adjustments were necessary. Each sample volume was measured individually (50–300 mL range) before the addition of a digestion solution at a final concentration of 5M  $\text{HNO}_3$ .

Samples were then subjected to thermal digestion at 80°C for 16–18 hours, allowing for gradual decomposition of natural organic material while minimizing potential polymer degradation. Following digestion, the samples were filtered onto pre-combusted quartz filters to retain microplastic residues. The filters were then placed in a drying oven until fully desiccated, ensuring the removal of residual moisture before further processing.

To enhance sample homogeneity and improve analytical reproducibility, the dried quartz filters underwent cryomilling using a Cryogenic Mill IQ MILL-2070 (Shimadzu Corporation, Kyoto, Japan). The cryomilling protocol involved loading the stainless-steel sample cup (cleaned and treated between each sample run) with one 10 mm tungsten grinding ball. Sufficient airspace within the cup was maintained to prevent sample overload and reduction in the grinding efficiency. The loaded sample cup was submerged in a liquid nitrogen bath for 5–8 minutes, ensuring adequate cooling before milling. The sample cup was then transferred to the milling chamber, where the following milling cycle was applied, with 20s of milling duration at 3000 rpm, followed by a 20s rest interval. The procedure was repeated to ensure the completeness of the milling and homogeneity of the sample.

After the milling process, the samples were allowed to warm to room temperature in a water bath, preventing condensation-related contamination. A measured 4–6 mg of the milled sample was weighed and transferred into sterilized eco-cups (Frontier PY1-EC80F) for subsequent pyrolysis-gas chromatography/mass spectrometry (Pyr-GC/MS) analysis.

### **2.3 Analysis**

Microplastic identification and quantification in the samples were conducted using pyrolysis-gas chromatography/mass spectrometry (Pyr-GC/MS). All sample preparation and analyses were performed under stringent contamination control measures to prevent exogenous plastic contamination. The analysis was conducted using a Shimadzu GCMS-TQ8040 system (Shimadzu Corporation) coupled with an EGA/PY-3030D Pyrolysis Unit (Frontier Labs). The system was configured with a UAMP column (40 m length, 0.25 mm i.d., 0.25  $\mu\text{m}$  film thickness) and a vent-free splitter.

Pyrolysis was performed in single-shot mode at 600°C, ensuring complete thermal degradation of polymeric materials. The GC/MS parameters (Table 1) included an inlet temperature of 300°C, MS source temperature of 250°C, and inlet pressure of 10.4 mL/min, with helium as the carrier gas. The column temperature program was as follows: an initial hold at 40°C for 3 minutes, ramping to 280°C at 20°C/min with a 10-minute hold, followed by an increase to 320°C at 40°C/min with a final hold of 15 minutes. The experiment was conducted in split injection mode with a split ratio of 1:50 to optimize resolution and sensitivity.

The polymer identification targeted twelve key microplastic polymers commonly detected in marine environments: polyethylene (PE), polypropylene (PP), polystyrene (PS), acrylonitrile-butadiene-styrene resin (ABS), styrene-butadiene rubber (SBR), polymethyl methacrylate (PMMA), polycarbonate (PC), polyvinyl chloride (PVC), polyurethane (PU), polyethylene terephthalate (PET), nylon-6 (N6), and nylon-66 (N66). The resulting pyrolysis products were compared against reference spectra to confirm polymer identity.

Calibration was conducted using a CaCO<sub>3</sub>-based microplastic calibration standard (Frontiers Labs), prepared at four different weights (0.4 mg, 2.00 mg, 4.00 mg, and 6 mg). Calibration curves were generated for each polymer, with R<sup>2</sup> values greater than 0.98 considered acceptable; any runs failing to meet this threshold were repeated to ensure analytical accuracy.

During mass spectrometry, pyrolysis products were ionized, and their mass-to-charge ratios (m/z) were measured to distinguish different molecular fragments. F-Search MPs software (version 3.7, Frontier Labs, Koriyama, Japan) was used for peak detection and spectral analysis, employing advanced algorithms to differentiate real signals from background noise. This ensured that only significant peaks corresponding to MP pyrolysis products were included in further analysis (Table 2). The relative peak heights of detected ions were compared against calibration standards to quantify MPs in the samples. Example spectra from an environmental plankton tow water sample are shown in Figures 4 and 5.

#### *Procedural Blanks*

A procedural blank was included in each digestion batch to ensure no contamination occurred during lab processing. The blank control was prepared for each batch under identical conditions as environmental samples. Digestion and filtration protocols were performed on combusted sampling containers and Quartz filters and subjected to py-GC/MS alongside environmental samples. No plastics were recovered in any of the procedural blanks, demonstrating that laboratory processing did not introduce any contamination into samples. The absence of plastic identification in blank samples provides confirmation that laboratory-induced cross-contamination was effectively prevented in the detection of plastics—highlighting the effectiveness of this method.

## **Results and Discussion**

### 3.1 A streamlined protocol for microplastic analysis in marine waters

In this project, we established a streamlined analytical protocol for MP analysis in marine waters, including sample collection, sample preparation, and sample analysis. We used plankton tow to collect water samples, which allowed the filtration of approximately 30,000 liters of water. This large volume of water collection ensures the representativeness of a sample considering that MPs in coastal waters are highly heterogeneous in terms of spatial distribution and polymer types (Wu et al., 2019). It should be noted that even though the plankton tow has a mesh size of 150  $\mu\text{m}$ , the MPs in the size of <150  $\mu\text{m}$  might have been collected if they were attached to larger natural particles or got attached during the towing process.

The key step of sample preparation is to remove the natural organic matter to a maximum extent while retaining the integrity of the plastics. This is essential for accurate Py-GC/MS detection of microplastics, as preliminary results show that undigested samples yielded fewer detected plastics compared to digested ones. As seen in Figures 6 and 7, interference from organic material in undigested samples overlapped with characteristic ion peaks, making it difficult to isolate plastic-specific fragments. The complex organic matrix in plankton tow samples masked key polymer signals, reducing analytical sensitivity. Digestion effectively removes organic interference, improving peak resolution and ensuring accurate polymer identification and quantification. These findings highlight the necessity of digestion in microplastic analysis.

We chose to use the digestion protocol of Tuttle and Stubbins (2023), in which the solution was composed of 70% nitric acid ( $\text{HNO}_3$ ) and sodium persulfate ( $\text{Na}_2\text{S}_2\text{O}_8$ ). While the oxidation power of this solution is much stronger than those used for biota analysis, such as potassium hydroxide (KOH) or enzymes (Karami et al, 2017 & von Friesen, 2019), it ensures that most, if not all, of the natural polymers get oxidized and thus ensures minimal organic interference, improving polymer identification via Py-GC/MS

We used nurdles of PE, PP, PS, PC and PET to examine the degree of potential oxidation to plastic samples. Our results showed that the nurdles mostly remained intact after the digestion based on SEM (Figure 8), suggesting a minimum degree of breakdown to the MPs in the samples. We did not evaluate the removal efficiency of natural organic matter with this acid solution, but the results of Tuttle and Stubbins (2023) showed that >99% of microalga is oxidized. Considering that plankton tow materials are dominated by marine biota, similar to microalga, we assume a similar digestion efficiency of >99%.

Even so, there was still a large quantity of residues left after the digestion, which might include inorganic minerals, resistant organic materials, and the plastics. It is unrealistic to analyze the whole Quartz filter (47mm diameter), due to the facts that the filter is too large to fit the pyrolysis cup (68 $\mu\text{L}$ ) and that the amount of plastics would oversaturate the mass spectrometer. It is challenging to homogenize a Quartz filter with the residues. We utilized a Cryogenic Mill

IQ MILL-2070 (Shimadzu Corporation, Kyoto, Japan) to grind the filter and optimize the protocol, as outlined in the Material and Method section. A picture of the residues is shown in Figure 9.

We chose Py-GC/MS, a state-of-the-art technique, for the identification and quantification of MPs in the filter samples (Ma et al., 2025). With high sensitivity, this technique can analyze MPs in trace levels, with detection limits ranging from 2-200 nanograms depending on specific polymer type (Marfella et al., 2024). There is also no need to isolate the plastics from the matrix before the analysis, as the pyrolysis will thermally decompose everything decomposable, and the mass spectrometry will select and analyze only the characteristic ion fragments that are from plastics. Because each polymer has its own characteristic ions and elution time in the GC column, one sample run will allow the quantification of all polymers based on the standards. Frontier Lab offers a composite standard containing 12 types of polymers in a matrix of calcium carbonate or silica, which further enhances the streamlined protocol of MP analysis.

### **3.2 Microplastic Composition Across Sites**

Microplastic concentrations varied across sites, with notable differences in both total polymer abundance and polymer type distributions. The highest concentration of microplastics was detected at CCB4, where total microplastic levels reached 1.63  $\mu\text{g/L}$ , while the lowest concentration was recorded at CCB3, with a total of 0.16  $\mu\text{g/L}$ . These findings suggest that microplastic contamination is not uniform across sampling locations, with certain sites exhibiting significantly higher accumulation (Tables 4-8). Such spatial variations are consistent with findings in other coastal systems, where hydrodynamics, industrial activity, and urban runoff contribute to localized microplastic hotspots (Morici et al., 2024). Compared to natural suspended particles, this concentration level of microplastics is much lower. For example, the amount of natural suspended particles is in the range of 2-4  $\text{mg/L}$  in the Mission Aransas Estuary, almost 3 orders of magnitude higher than the MPs. However, considering that MPs are resistant to environmental degradation and the potential of being bioaccumulated in biota and humans, this trace level of MPs is a concern in terms of ecosystem health.

Among individual polymers, PE (Polyethylene) exhibited the highest concentration at 1.14  $\mu\text{g/L}$  at CCB1, while ABS (Acrylonitrile Butadiene Styrene) was the least abundant, recorded at only 0.00012  $\mu\text{g/L}$  also at CCB1. The overall range of individual microplastic concentrations spanned from 0.00012  $\mu\text{g/L}$  to 1.14  $\mu\text{g/L}$ , indicating significant variability in polymer distribution across the study area. These findings align with recent studies suggesting that polyolefin-based plastics, such as PE and PP, are the dominant pollutants in marine environments due to their extensive use in packaging and industrial applications, or high production (Kesavan et al., 2025; Vijayaprabakaran et al., 2024).

A comparison of polymer compositions across sites revealed both similarities and differences in microplastic profiles. PE, PP, and PET were the most dominant polymers across all sampling sites when measured by presence, suggesting that polyolefin-based plastics are widespread in the Texas Coastal Bend (Figure 10). This is consistent with the facts that these are the dominant types of plastics in the production and our daily use, and that these are also the commonly found MPs in other aquatic environments (Koelmans et al., 2019). However, our results also showed that certain locations exhibited unique polymer distributions. For example, CCB4 was the only site to detect polyvinyl chloride (PVC), while CCB1 showed a distinct increase in PET. Despite only being detected at one location, PVC was the third most prevalent polymer type per liter after PE and PP when measured by total quantity. These variations likely reflect differences in local plastic sources, such as industrial discharges, urban runoff, or specific land-use activities (Rossi et al., 2025). Such site-specific polymer variations emphasize the influence of land-based inputs, particularly from urbanized and industrialized regions, on coastal plastic pollution. Several studies have highlighted that microplastic accumulation patterns are often dictated by proximity to major urban centers and industrial outflows, with estuarine and coastal zones acting as critical sinks for plastic waste (Grillo et al., 2025). The presence of PVC at a single site may be linked to construction runoff or wastewater discharge, as PVC is commonly used in building materials and piping (Boshoff, 2024). This result also demonstrates the need for time-series data to get insights into the sources and variations in season, which for example will confirm whether the presence of PVC is due to a point source release or a transient phenomenon. Despite these differences, some patterns remained consistent. PE and PP were the dominant polymers at nearly all locations, reinforcing their prevalence in marine environments due to their buoyant nature and persistence in seawater (Amarathunga et al., 2025). Less abundant polymers, such as styrene-butadiene rubber (SBR), nylon-66 (N66), and polymethyl methacrylate (PMMA), were detected at lower concentrations but were present at multiple sites, indicating a more diffuse contribution of secondary microplastics from various sources. These secondary microplastics likely originate from tire wear, textile fibers, and weathered consumer plastics, which aligns with findings in other estuarine environments (Rabari et al., 2024).

These findings underscore the importance of spatial monitoring in understanding microplastic pollution dynamics. While certain locations exhibit significantly elevated contamination, all sampled sites contained detectable microplastics, highlighting the pervasive nature of plastic pollution in coastal ecosystems. Given the variability in polymer types and concentrations, future investigations should focus on identifying the sources contributing to site-specific polymer variations. Further refinement of sampling methodologies, including standardized net mesh sizes and water volume collection strategies, may also improve comparability across studies (Li et al., 2024). Targeted mitigation strategies, such as improved waste management policies and plastic reduction initiatives, are crucial to reducing microplastic inputs into marine environments (Ovide et al., 2025).

### **3.4 Scaling to a Bay System: Estimating Microplastic Concentrations in Water**

To evaluate the broader environmental implications of our findings, we applied a scaling approach to estimate the total polyethylene (PE) load in Corpus Christi Bay, where we have the relatively complete data. By extrapolating our measured microplastic concentrations to the entire bay system, we aimed to approximate the total burden of this dominant polymer type in the ecosystem. Given that Corpus Christi Bay has an estimated volume of 1.35 trillion liters ( $1.35 \times 10^{12}$  L), this concentration scales to a total estimated 918 kg (0.92 metric tons) of polyethylene.

We acknowledge that this estimation may suffer a large uncertainty, given the high heterogeneity of MPs in marine waters both spatially and temporally. However, the large volume of water processes, at about  $30 \text{ m}^3$ , and the direct quantification of MPs using Py-GC/MS both increase the reliance of the calculation, when compared to other works that are based on the number of plastic particles, sometimes particles that are suspected to be plastics (Conkle, 2018; Koelmans et al., 2019), but might be not. These calculations highlight the potential magnitude of microplastic pollution in coastal bay systems, emphasizing the need for expanded spatial assessments to capture local variability. Prior research has shown that microplastic transport and deposition patterns in estuarine environments are influenced by hydrodynamic conditions, proximity to urban and industrial outflows, and interactions with sediment and biological uptake (Grillo et al., 2025; Li et al., 2024). Given the dominance of PE and PP polymers in global marine pollution, similar scaling approaches have been applied in other coastal environments, reinforcing the need for standardized methodology in microplastic quantification at larger spatial scales (Rossi et al., 2025).

This method of upscaling localized microplastic concentrations to a bay-wide estimate provides a valuable tool for monitoring strategies and policy decisions regarding microplastic pollution. Several studies emphasize that microplastics act as vectors for chemical pollutants, with potential ecological risks for filter feeders, fish, and other marine organisms (Ovide et al., 2025). Additionally, as plastic production and waste mismanagement continue to rise, scaling approaches like this can help establish baseline data for assessing long-term trends and evaluating the effectiveness of pollution mitigation strategies.

While this estimate provides an initial assessment of PE loads, further research is needed to refine these calculations by accounting for seasonal and hydrodynamic variability, which may influence microplastic dispersion across different regions of the bay. A more detailed analysis could also distinguish between various sources of microplastics, such as land-based runoff, atmospheric deposition, and maritime activities, to better understand their origins. Moreover, integrating advanced sampling techniques, such as automated water filtration and remote sensing, would enhance the accuracy of large-scale quantification efforts.

Ultimately, our findings reinforce the pervasive nature of microplastic pollution and highlight the importance of comprehensive monitoring frameworks that integrate localized sampling with broad-scale modeling approaches. Future studies should adopt an interdisciplinary approach,

combining oceanographic modeling, chemical characterization, and biological impact assessments to develop a more holistic understanding of the long-term ecological consequences of microplastic contamination in estuarine environments.

## **Conclusions**

In this project, we established a streamlined protocol of microplastic analysis in natural waters, including the well-thought-out steps from water collection, sample digestion and plastic quantification. In the water collection, we took advantage of the large volume filtration capacity in plankton tow (150  $\mu\text{m}$  mesh size), which processed about 30  $\text{m}^3$  of seawater; this large volume maximized the representativeness of the sample. To remove the natural organic matter in the sample, an essential step to concentration and purify the plastics before the analysis, we adopted a chemical cocktail of nitric acid and sodium persulfate with enough oxidation power to destroy the natural polymers while leaving the plastics intact, based on test results. We utilized the state-of-the-art analytical instrument in pyrolysis coupled with gas chromatography mass spectrometry, which offers high sensitivity for trace levels of plastics and can quantify multiple types of plastic polymers in a single sample run. This protocol has been proven to be feasible, reliable and sensitive for microplastic analysis in natural waters, and is much more superior than other protocols in terms of quantification, such as those relying on microscopy or micro-FTIR etc.

As the first result of such, we showed that concentrations of microplastics varied widely in the Corpus Christi Bay, ranging from 0.001 – 1.6  $\mu\text{g/L}$  among the sampling sites. The plastics were dominated by polyethylene (PE), polypropylene (PP) and polyethylene terephthalate (PET), together with styrene-butadiene rubber (SBR), nylon-66 (N66), and polymethyl methacrylate (PMMA) as the minor components. As a simple extrapolation from these results, the whole Corpus Christi Bay may contain up to about 1 metric ton of PE. While the analysis and further sampling are still ongoing, this work offers the first direct quantification of microplastics of the bays and estuaries in Texas Coastal Bend, or any other natural waters for this matter. These data, including those that will be collected in Phase II of the project, will help policy makers evaluate the source and degree of the plastics contamination and the potential ecological impact, and make appropriate action or policies to maintain healthy estuarine ecosystems.

## **Acknowledgement**

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## Figures and Tables

Table 1 Pyrolysis and Gas chromatography mass spectroscopy settings

Microfurnace Pyrolyzer	EGA/PY-3030D Pyrolysis Unit
Mode	Single Shot
Carrier Gas	Helium
Temperature	600 °C
Pyrolysis Time	0.3 mins (pyrolysis) 40 mins total run time
Transfer Line Temperature	300° C
Gas Chromatograph	Shimadzu GCMS-TQ8040
Injector	50:1
Mode	Split
Temperature	300° C
Column	Ultra Alloy Microplastics (UAMP) Column kit specifically designed for microplastics analysis (UA5-40M-0.5F, Frontier Labs, Koriyama, Japan)
Flow (Const.) or pressure (cons)	14.3 psi
Temperature Program	40 °C hold for 2 minutes Ramp at 20° C/min to 280 °C hold for 10 minutes Ramp 40 °C/min to 320° C hold for 15 min
Transfer Line Temperature	280 °C
Mass Spectrometer	
Ionization Energy	69.9 (70eV)
Scan Rate	3.42 scan/s
Scan Range	30-450

Table 2 Pyrolyzates used for identification and quantification of 12 polymers types

<b>Polymer of Interest</b>	<b>Extracted-Ion Chromatogram Spectrum (EIC)</b>
Polyethylene (PE)	m/z: 82 1,20- Heneicosadine (C21")
Polypropylene (PP)	m/z: 126 2,4-Dimethyl-1-heptene (C9')
Polyvinyl Chloride (PVC)	m/z: 128 Naphthalene (Naph)
Polyethylene Terephthalate (PET)	m/z: 182 Benzophenone (BP)
Polystyrene (PS)	m/z: 91 Styrene Trimer (SSS)
Polycarbonate (PC)	m/z: 134 4-isopropenylphenol (IPP)
Polyurethane (PU)	m/z: 198 4,4'-Methylenedianiline (MDA)
Styrene Butadiene Rubber (SBR)	m/z: 104 4-Phenylcyclohexene (SB)
Polymethyl Methacrylate (PMMA)	m/z: 100 Methyl Methacrylate (MMA)
Acronitrile Butadiene Styrene Resin (ABS)	m/z: 170 2-Phenethyl-4-phenylpent-4-enenitrile (SAS)
Nylon 6 (N6)	m/z: 113 $\epsilon$ -Caprolactam (Capro)
Nylon 66 (N66)	m/z: 84 Cyclopentanone (CP)

Table 3. Water quality parameters in Corpus Christi Bay across 5 sites.

Site	Water Quality Parameter	Measurements
CCB1	Specific conductivity	60.81 mS/cm
	pH	7.97
	Dissolved oxygen (DO)	5.82 mg/L
	Salinity	35.22
CCB2	Specific conductivity	60.89 mS/cm
	pH	8.03
	Dissolved oxygen (DO)	6.06mg/L
	Salinity	35.17
CCB3	Specific conductivity	62.14 mS/cm
	pH	8.03
	Dissolved oxygen (DO)	5.41 mg/L
	Salinity	36.39
CCB4	Specific conductivity	63.68mS/cm
	pH	8.13
	Dissolved oxygen (DO)	6.04mg/L
	Salinity	36.36
CCB5	Specific conductivity	63.74mS/cm
	pH	8.12
	Dissolved oxygen (DO)	6.12mg/L
	Salinity	36.54

Table 4. Quantification of plastics in Corpus Christi Bay site 1 (CBB1). Averaged for duplicates

Polymer	Quantity [ $\mu\text{g/L}$ ]
PE	1.143831
PP	0.190507

PET	0.007007
ABS	0.000123

Table 5. Quantification of plastics in Corpus Christi Bay site 2 (CBB2). Averaged for duplicates.

Polymer	Quantity [ $\mu\text{g/L}$ ]
PE	0.6252948265
PP	0.178725245
PET	0.004423421
PS	0.001696361
N66	0.001295533
PC	0.000416605
ABS	0.0002726395

Table 6. Quantification of plastics in Corpus Christi Bay site 3 (CBB3). Averaged for Duplicates

Polymer	Quantity [ $\mu\text{g/L}$ ]
PE	0.082858727
PP	0.054077255
PMMA	0.011867406



PET	0.004711261
N66	0.002269132
PC	0.000578245
ABS	0.0002061635

Table 7. Quantification of plastics in Corpus Christi Bay site 4 (CBB4). Averaged for Duplicates

Polymer	Quantity [ $\mu\text{g/L}$ ]
PE	1.1315736945
PP	0.2616543485
PVC	0.21652541
PET	0.01032534
PMMA	0.002734139
ABS	0.001788533
PC	0.001085844

Table 8. Quantification of plastics in Corpus Christi Bay site 5 (CBB5). Averaged for Duplicates

Polymer	Quantity [ $\mu\text{g/L}$ ]
PE	0.414548946
PP	0.049174712
PET	0.004161658
PMMA	0.00075885

PC	0.000391778
ABS	0.000215225

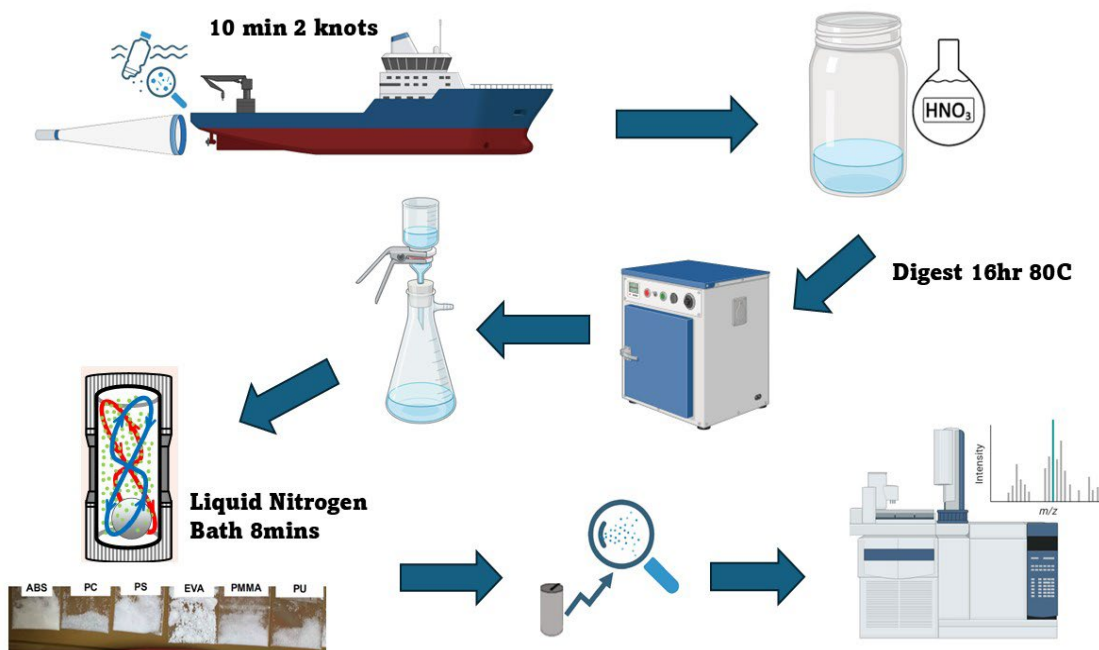


Figure 1. Graphical representation of sampling protocol

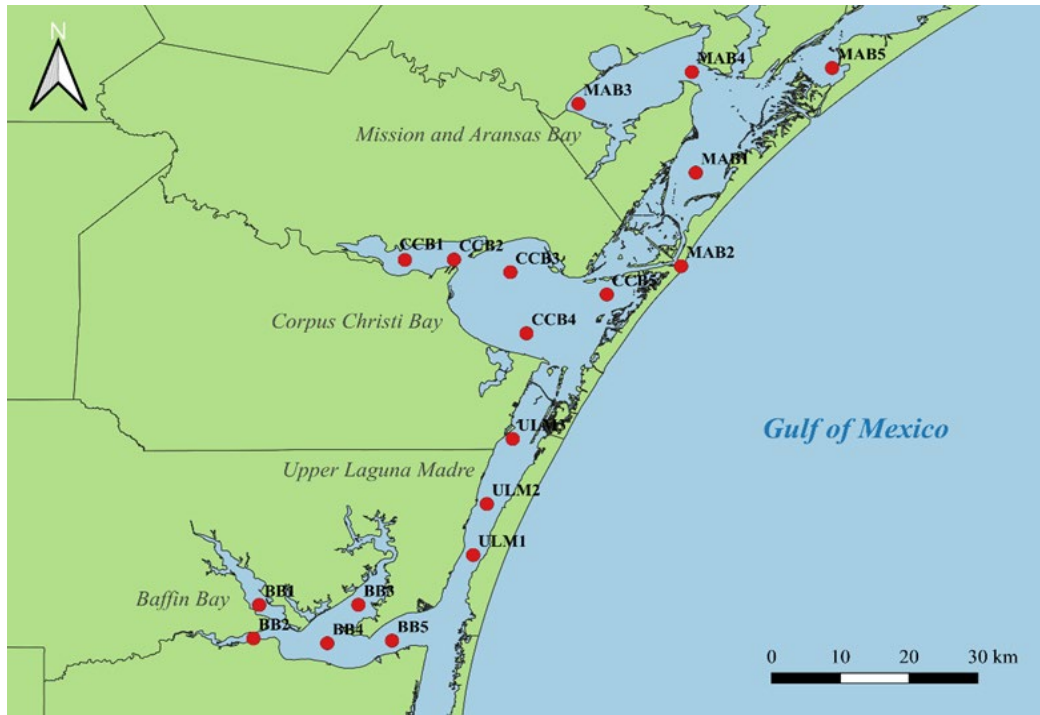


Figure 2. Sampling stations at the three estuarine systems: Copano, Aransas and Mesquite bays, Neuces and Corpus Christi bays, and Upper Laguna Madre and Baffin Bay. There are 18 stations where microplastic will be collected.



Figure 3. Jellyfish excluder added to bongo plankton net

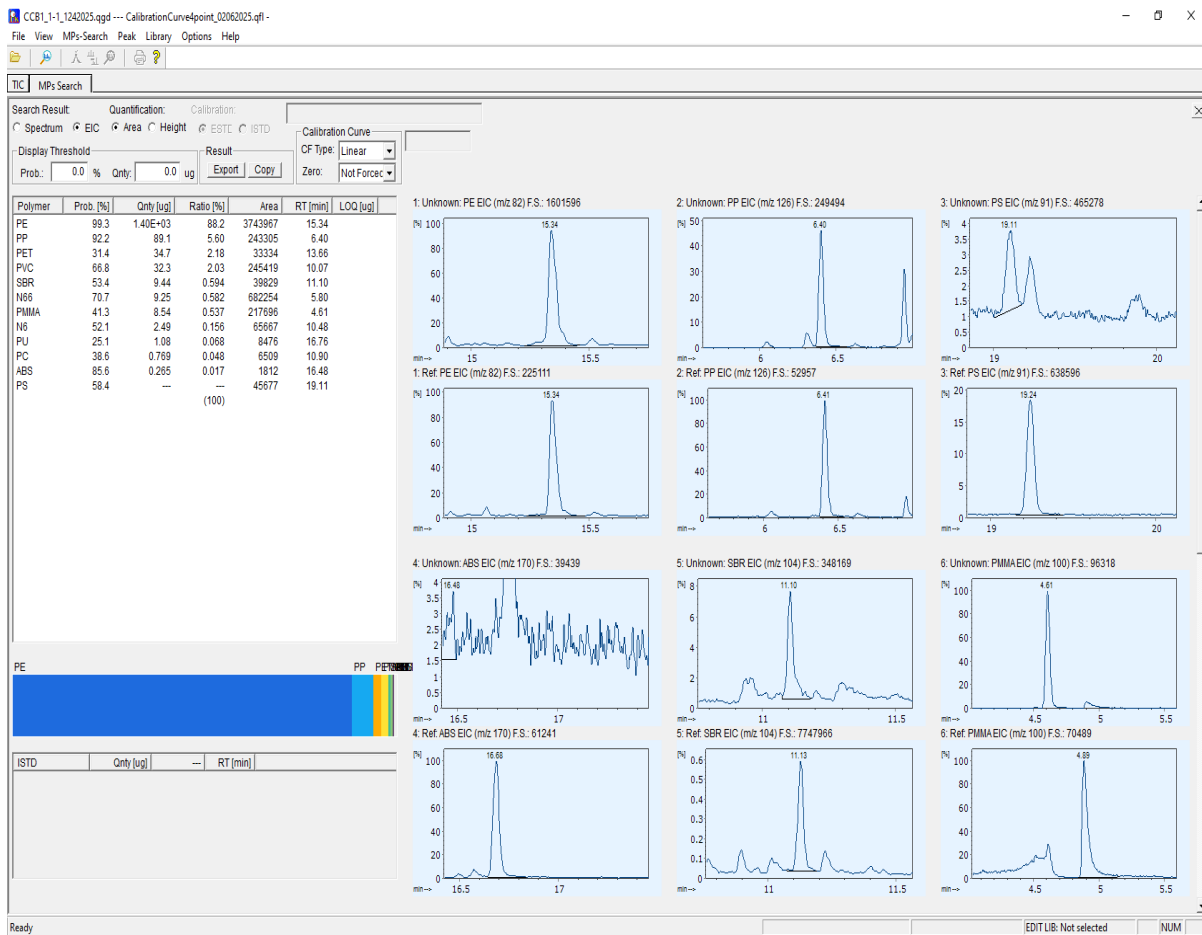


Figure 4. F-search results of Corpus Christi Bay site 1. Displays percentages of likely polymer identification with quantitative measurements given in micrograms

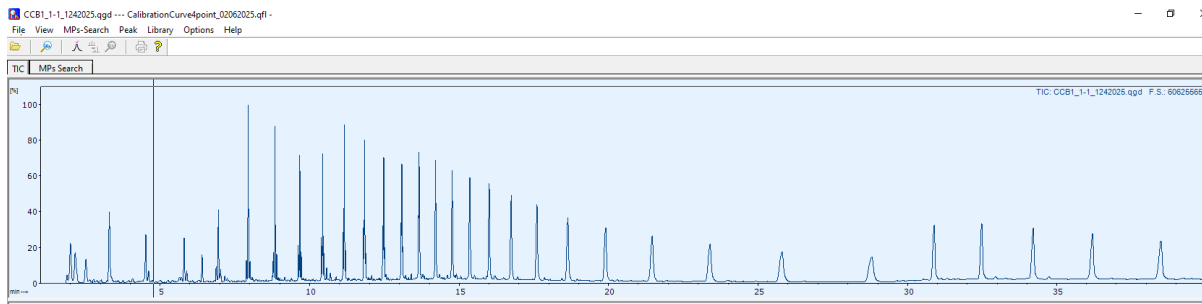


Figure 5. TIC of environmental sample Corpus Christi Site 1

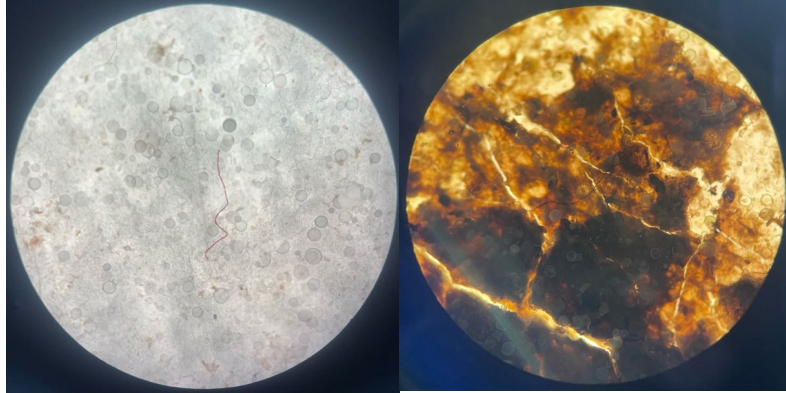


Figure 6. Differences in filter appearance in relation to digestion methods. (a) Digested filter with removal of organic matter (b) undigested filter.

### MICROPLASTIC COUNT IN UNDIGESTED AND DIGESTED SAMPLES PER SITE

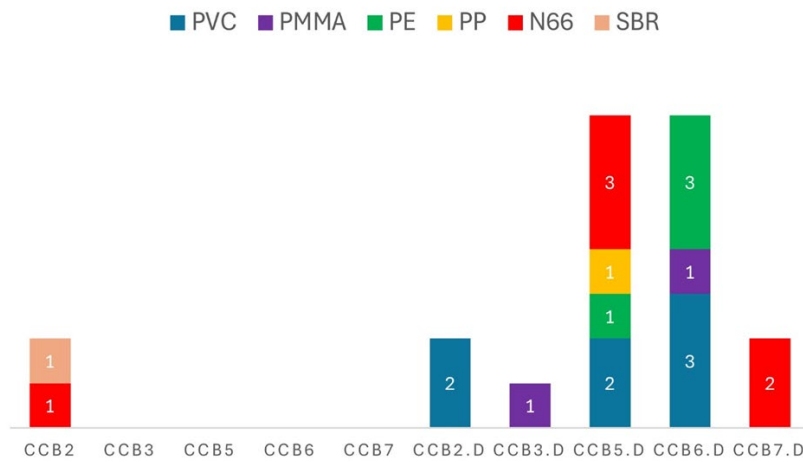


Figure 7. Preliminary results regarding the qualification and counts of MPs in Corpus Christi Bay, Texas. Prior to the new subsampling method with cryomill. Sites with “D” indicate digested samples while the absence of “D” refers to undigested.

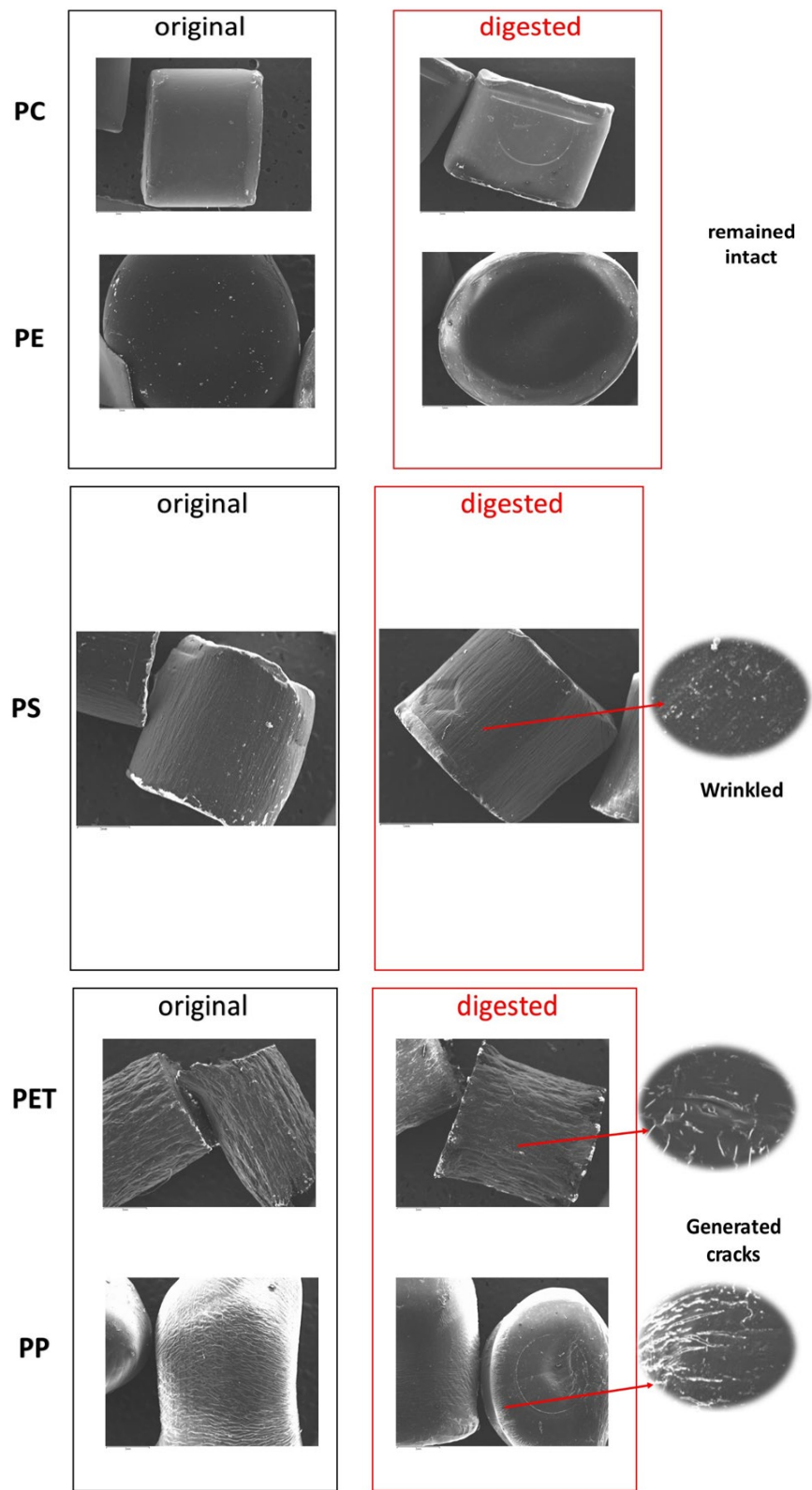


Figure 8. SEM images of 5 plastic nurdle pellets prior to and following exposure to digestion protocol. Mass recoveries were not extracted from this data.



Figure 9. Cryogenic mill samples

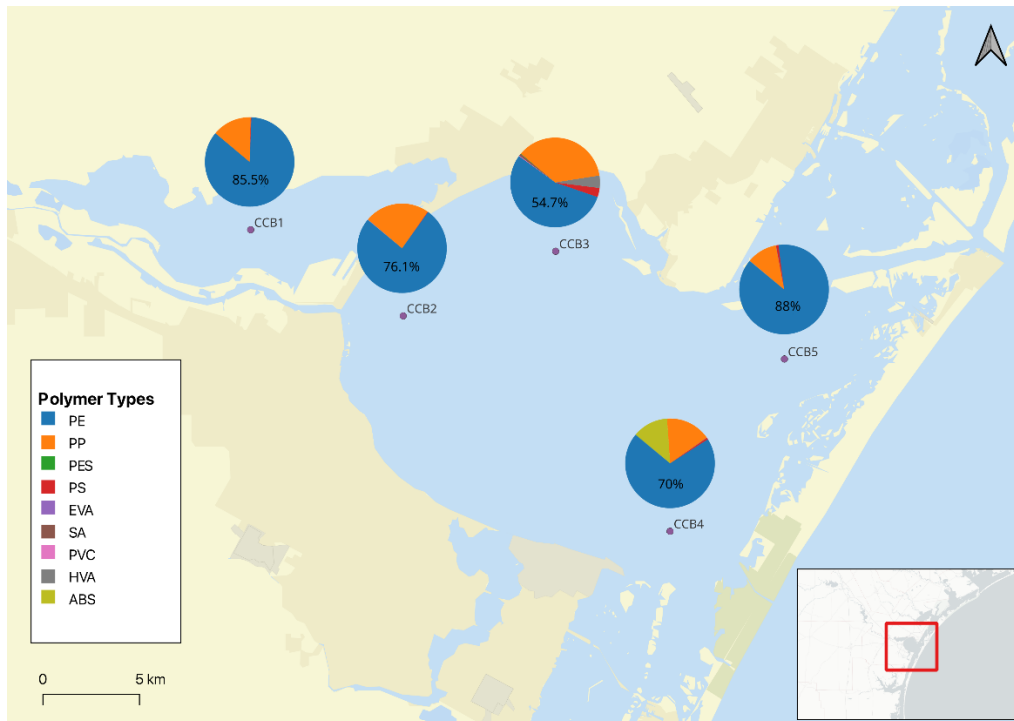


Figure 10. Relative proportion of polymer types by site for Corpus Christi Bay