EEET ECOLOGICAL ENGINEERING & ENVIRONMENTAL TECHNOLOGY

Ecological Engineering & Environmental Technology, 2026, 27(5), 430–445
https://doi.org/10.12912/27197050/220346
ISSN 2719–7050, License CC-BY 4.0

Received: 2026.03.20
Accepted: 2026.04.15
Published: 2026.05.01

Microplastics in marine sediments from Burgas Bay, Black Sea: Abundance, polymer composition, and comparison of sodium chloride and magnesium chloride density separation methods using µFTIR imaging

Sevdalina Turmanova[1], Dimitrina Stoyanova Kiryakova[1*], Emiliya Ivanova[2], Plamena Atanasova[1], Ganka Kolchakova[1], Antoniya Ilieva[3], Elena Mollova[2], Yancho Hristov[1]

[1] Department of Materials Science, Burgas State University „Prof. Dr. Assen Zlatarov", Y. Yakimov Str. 1, Burgas 8010, Bulgaria
[2] Department of Ecology and Environmental Protection, Burgas State University „Prof. Dr. Assen Zlatarov", Y. Yakimov Str. 1, Burgas 8010, Bulgaria
[3] Department of Chemical Technologies, Burgas State University „Prof. Dr. Assen Zlatarov", Y. Yakimov Str. 1, Burgas 8010, Bulgaria
* Corresponding author's e-mail: dimitrina-kiryakova@uniburgas.bg

ABSTRACT

Microplastics are increasingly detected in marine sediments, where they can accumulate over time. The aim of this study was to determine the abundance and polymer composition of microplastics in sediments from Burgas Bay, western Black Sea. Sediment samples were collected from six stations across three coastal areas with differing anthropogenic influence: Burgas Beach, Chengene Skele, and St. Anastasia Island. Microplastics were extracted using density separation with two flotation solutions (NaCl and $MgCl_2$), followed by filtration and µFTIR imaging spectroscopy. Particle abundances ranged from 0.0 to 5497.8 MPs kg^{-1} dry sediment depending on the station and extraction solution. $MgCl_2$ generally yielded higher particle counts than NaCl, although higher values were observed with NaCl at some stations, indicating that extraction efficiency may depend on particle density and sediment characteristics. Nine polymer types were identified, with polyamide (48.6%), polypropylene, polyethylene, and ethylene vinyl alcohol being the most frequent. These findings provide detailed quantification and polymer characterization of microplastics in Burgas Bay sediments, extending previous regional observations and offering targeted data for future monitoring and comparative studies in the Black Sea region.

Keywords: microplastic pollution, sediment contamination, polymer identification, µFTIR spectroscopy, coastal environment, density separation.

INTRODUCTION

Microplastics (MPs) pollution of aquatic ecosystems has become one of the most significant environmental problems in recent decades. Due to their small size and high resistance to degradation, microplastics accumulate in various environmental compartments, including the water column, biota and sediments. Their presence has been reported in a wide range of marine environments, from coastal and estuarine zones to deep-sea sediments, indicating that plastic particles can be transported and accumulated over large spatial scales (Woodall et al., 2014; Van Cauwenberghe et al., 2015).

In addition to being physical pollutants, MPs can interact with various chemical compounds in the environment. Their surfaces facilitate the adsorption of pollutants such as heavy metals and persistent organic compounds, which may affect

their mobility and bioavailability (Rochman et al., 2013; Holmes et al., 2012). As a result, microplastics may act as potential vectors for the transport of toxic substances in marine ecosystems. The ingestion of microplastic particles by marine organisms, ranging from invertebrates to fish, has also been documented, raising concerns about their transfer through food webs and the associated ecological risks (Cole et al., 2011; Wright et al., 2013). The Black Sea remains relatively poorly studied in terms of microplastic distribution compared to other marine basins. As a semi-enclosed sea with limited water exchange with the Mediterranean Sea, it creates conditions that may favor the retention and accumulation of pollutants, including plastic particles (Cincinelli et al., 2021; Berov and Klayn, 2020). Despite the increasing number of studies on microplastics in marine environments, information on their distribution in sediments from the Bulgarian Black Sea coast remains limited. Burgas Bay is one of the most intensively used coastal areas in the western Black Sea and is subject to significant anthropogenic pressures associated with port activities, tourism, fishing and urbanization. These activities may contribute to the introduction of plastic waste into the coastal zone and its subsequent accumulation in sediments. Available studies in the Black Sea indicate that microplastics are present in both coastal and deeper sediments, highlighting the importance of bottom sediments as a sink for plastic particles (Cincinelli et al., 2021; Bobchev et al., 2024).

The study of microplastics in environmental matrices remains methodologically challenging, as the accuracy and comparability of results strongly depend on the analytical workflow applied. In particular, sediment samples represent a complex matrix, where the efficiency of microplastic extraction is influenced by multiple factors, including particle size, polymer density, and the presence of organic matter. As a result, inconsistencies in sampling strategies, extraction procedures, and analytical techniques continue to hinder the comparability of data across studies and regions (Hidalgo-Ruz et al., 2012; Prata et al., 2019; Shim et al., 2018).

Among the critical steps in microplastic analysis, density separation is widely recognized as a key procedure for isolating plastic particles from mineral sediments. However, the choice of flotation solution remains insufficiently standardized, and different solutions may vary significantly in their efficiency to recover polymers with contrasting densities. While commonly used solutions such as NaCl are cost-effective and environmentally safer, their relatively low density may limit the recovery of high-density polymers. In contrast, alternative solutions, including magnesium-based salts, offer higher density but remain less frequently evaluated under comparable environmental conditions (Claessens et al., 2013; Imhof et al., 2012).

Despite the growing number of studies addressing microplastic contamination in marine sediments, the Black Sea – and particularly the Bulgarian coastal zone – remains underrepresented in terms of methodologically consistent and polymer-specific investigations. Existing studies have primarily focused on the occurrence and distribution of microplastics, whereas limited attention has been paid to how methodological choices influence the reported abundance and composition of particles. This lack of methodological comparability represents a critical gap, especially for semi-enclosed basins such as the Black Sea, where pollutant accumulation processes may differ substantially from those in open marine systems.

To the best of our knowledge, this is among the first studies for the Bulgarian Black Sea coast to combine μFTIR-based polymer identification with a comparative evaluation of NaCl and $MgCl_2$ density separation solutions for marine sediment samples.

In this context, a systematic evaluation of extraction efficiency using different density separation solutions is essential for improving the reliability and comparability of microplastic data in marine sediments. Furthermore, the integration of μFTIR imaging enables a more precise characterization of polymer composition, allowing for a better understanding of how methodological factors influence not only particle recovery but also the detected polymer spectrum.

Therefore, the aim of the present study was to comparatively evaluate the efficiency of two density separation solutions (NaCl and $MgCl_2$) for the extraction of microplastics from marine sediments collected in Burgas Bay (Black Sea), and to assess their influence on the abundance, spatial distribution, and polymer composition of recovered particles using μFTIR imaging spectroscopy. It was hypothesized that the higher-density $MgCl_2$ solution would enhance the recovery efficiency of microplastics, particularly for polymers

with densities exceeding that of NaCl solutions. Additionally, it was expected that the choice of flotation medium would influence the observed polymer distribution, thereby affecting the interpretation of microplastic contamination patterns in marine sediments.

MATERIALS AND METHODS

Study area

The Burgas Bay is located in the western part of the Black Sea. It is one of the largest semi-enclosed coastal areas in Bulgaria and is affected by various anthropogenic activities, including urbanization, port activities, tourism and fishing. The bay is located near the city of Burgas and includes a variety of coastal and marine habitats. Due to the intense human impact, the area is considered a potential area for the accumulation of anthropogenic pollutants, including microplastics in marine sediments. Within the framework of this study, six sampling stations were selected, located in different parts of Burgas Bay. The location of the stations is shown in Figure 1.

Characteristics of sampling points

Sediment samples were collected from six stations located in three different areas of Burgas Bay, characterized by varying degrees of anthropogenic impact. Two stations (S1 and S2) were located in the area of Burgas Beach, which is a popular tourist area located in close proximity to the city coastline and the port of Burgas. Two stations (S3 and S4) were located in the area of Chengene Skele, a fishing village where fishing activities are carried out and small vessels moor. The remaining two stations (S5 and S6) were located near the island of St. Anastasia, which is located in the southern part of the Burgas Bay and represents a relatively less urbanized area. The selection of these three areas allows for an assessment of the spatial distribution of microplastics in the sediments of the Burgas Bay, depending on the different potential sources of anthropogenic pollution. The geographical coordinates and water depth at the sampling points are presented in Table 1. The sampling depth varies between 2.8 and 7.3 m.

Sediment sampling

Marine sediment samples were collected from six stations (S1–S6) located in the Burgas Bay, Black Sea, during the summer season (June–August 2025). Mechanical samplers, such as the Van Veen sampler, are widely used to collect bottom sediments, which allow for the collection of representative samples with minimal

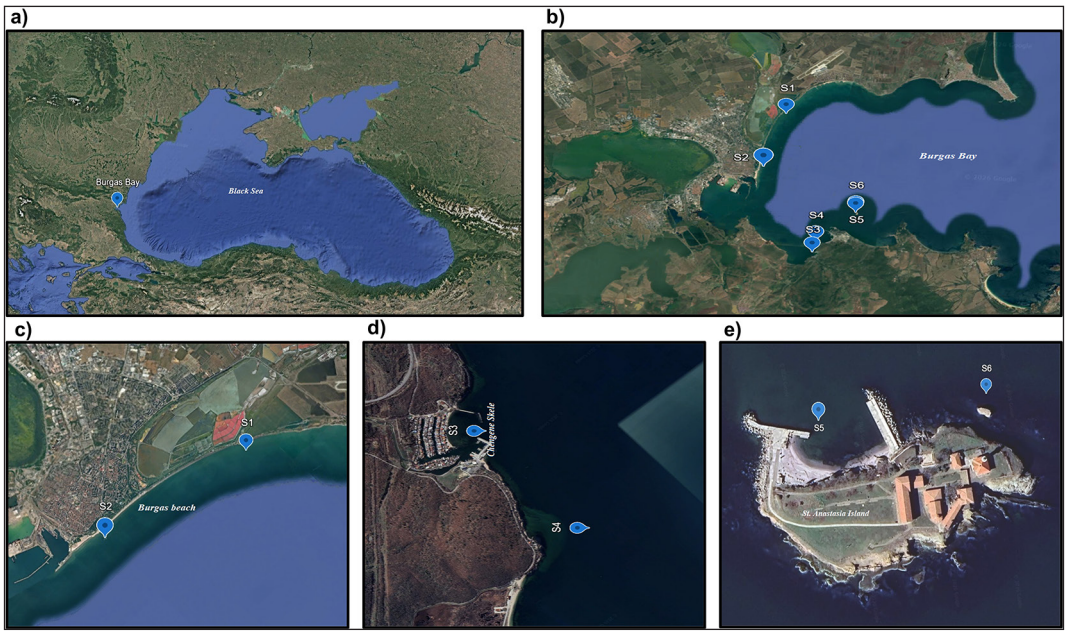

Figure 1. Study area and sediment sampling stations (S1–S6) in Burgas Bay, Black Sea: (a) regional map of the Black Sea showing the study area; (b) detailed map of Burgas Bay with the distribution of sampling stations; (c) Burgas Beach (S1–S2); (d) Chengene Skele (S3–S4); (e) St. Anastasia Island (S5–S6)

Table 1. Sampling stations and geographic coordinates in Burgas Bay

Site	Area	Sampling location	Latitude (°N)	Longitude (°E)	Depth (m)
S1	Burgas Beach	Burgas pier	42°31'58.78"	27°29'51.47"	4.8
S2	Burgas Beach	Burgas salt pools	42°29'42.52"	27°29'12.62"	5.0
S3	Chengene Skele	Harbor entrance	42°26'16.64"	27°31'35.32"	6.0
S4	Chengene Skele	Dalyana	42°26'45.19"	27°31'40.65"	5.1
S5	St. Anastasia Island	Island pier	42°28'02.38"	27°33'08.67"	2.8
S6	St. Anastasia Island	Mushroom rock	42°28'06.20"	27°33'08.70"	7.3

disturbance of the sediment layer structure (Van Cauwenberghe et al., 2015). Sampling was performed using a Van Veen grab sampler (sampling area ~0.1 m^2) launched from a research boat. Sampling was conducted at water depths ranging from 2.8 to 7.3 m, depending on the station, as presented in Table 1. The selected depth range represents shallow coastal marine sediments influenced by both natural processes and anthropogenic activities. The upper sediment layer (approximately 0–5 cm) was carefully collected, representing recently deposited material and the most relevant fraction for microplastic accumulation. This layer is commonly analyzed in microplastic studies, as it reflects recent deposition and ongoing environmental inputs. At each station, one composite sample (~500 g wet weight) was obtained. After retrieval, the sediment was transferred using a metal spoon into pre-cleaned metal containers with lids to minimize contamination. All sampling equipment was rinsed with ambient seawater between stations. Samples were transported to the laboratory in a cooled transport container and stored at 4°C until further processing. Prior to microplastic extraction, the sediment samples were air-dried at ambient temperature and homogenized. The mass of dry sediment used for microplastic extraction was 50 g for each sample. The sediment sampling procedure and sample handling are illustrated in Figure 2.

In this context, sediment samples were processed using a combined analytical approach involving sequential physical and chemical processing steps. The procedure included drying and sieving, density separation using sodium and magnesium chloride solutions, removal of organic matter, and vacuum filtration to isolate MPs on a filter membrane (Claessens et al., 2013; Nuelle et al., 2014). The extracted particles were subsequently analyzed by μFTIR microscopy, which enables reliable determination of the polymer composition based on the characteristic infrared spectra (Löder and Gerdts, 2015; Primpke et al., 2017).

Microplastics extraction

Microplastics were extracted from sediment samples using a density separation method. Exactly 50 g of dried sediment was placed in a 250 mL glass cylinder, and 100 mL of saturated salt solution was added. Two flotation solutions were used: sodium chloride (NaCl, $\rho \approx 1.20$ g cm^{-3}) and magnesium chloride ($MgCl_2$, $\rho \approx 1.26$ g cm^{-3}). The mixture was thoroughly mixed to ensure proper dispersion of particles. Subsequently, bubbling was applied for 1 h at room temperature using an air pump providing continuous airflow through a flexible tube (hose) inserted into the suspension. After bubbling, the samples were left undisturbed for 24 h to allow complete sedimentation of the mineral fraction. The supernatant containing floating particles was carefully decanted to avoid disturbing the settled sediment. The collected supernatant was treated with 30% hydrogen peroxide (H_2O_2, 20 mL) to remove organic matter. Oxidation was carried out under constant stirring (400 rpm) for 12 h at room temperature. The extraction procedure was performed once per sample due to the relatively low organic content of the sediment and based on previous method validation (Turmanova et al., 2026). For each sample, a single extraction procedure was performed for each flotation solution. Although repeated extractions can improve recovery efficiency, they were not applied in the present study. Although NaCl and $MgCl_2$ solutions are widely used for density separation of microplastics, their density may not be sufficient for complete recovery of high-density polymers such as PET and PVC. Therefore, the abundance of these polymers may be underestimated in the present study. The overall

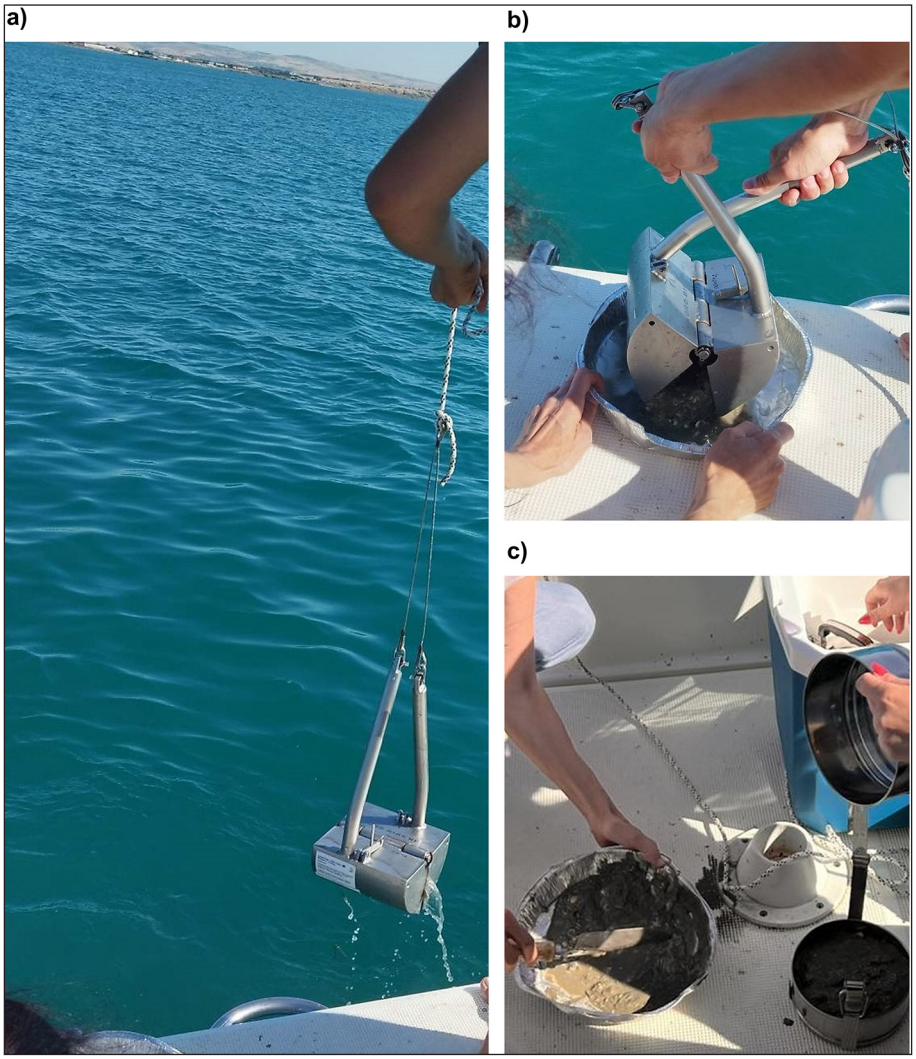

Figure 2. Sediment sampling procedure in Burgas Bay, Black Sea, at water depths between 2.8 and 7.3 m: (a) deployment of the Van Veen grab sampler; (b) retrieval of the upper sediment layer after sampling; (c) transfer of sediment into pre-cleaned metal containers

workflow of microplastic extraction and subsequent analysis is presented in Figure 3.

Filtration and drying

The resulting supernatant was filtered using a vacuum filtration system equipped with Anodisc filters (diameter 25 mm; pore size 0.2 µm). Filtration was performed under constant vacuum pressure (~0.5 bar). Filters were handled using metal tweezers and placed in covered glass Petri dishes to prevent contamination. After filtration, the filters were rinsed with distilled water and dried at 40 °C for 12 h. The main steps of the laboratory procedure, including filtration and sample preparation, are illustrated in Figure 4. The study was conducted using one sediment sample per station and one extraction per flotation solution. Therefore, the obtained values should be considered indicative for the investigated sampling period and location. Due to the absence of biological and technical replicates, the variability of microplastic abundance could not be statistically assessed.

Identification of microplastics

The identification of microplastic particles is based on the determination of their morphological and chemical characteristics by analytical techniques such as optical microscopy, Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy, which allow the identification of the polymer composition based on the characteristic spectral signatures (Hidalgo-Ruz et al., 2012; Shim et al., 2018). µFTIR microscopy is

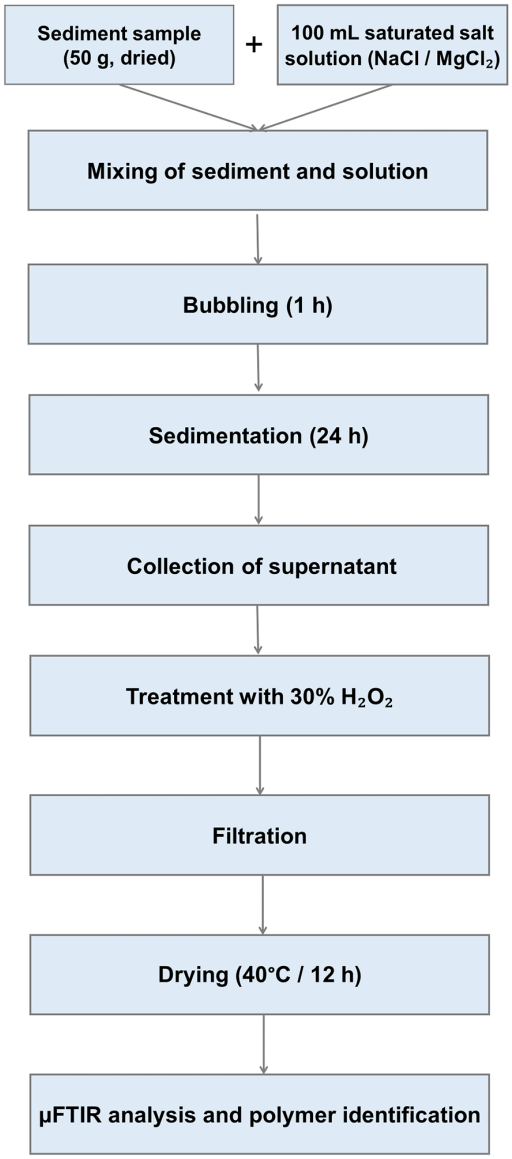

Figure 3. Workflow of microplastic extraction and analysis from marine sediments, including density separation, oxidative treatment, filtration, and µFTIR identification

particularly widely used, providing high spatial resolution and the possibility of automated mapping and identification of polymers in the micrometer range (Löder and Gerdts, 2015; Primpke et al., 2018). Micro-Fourier Transform Infrared spectroscopy (µFTIR) was used to identify polymer particles. Spectral imaging was performed using a LUMOS II microscope (Bruker) equipped with a focal plane array (FPA) detector with a 32 × 32 pixel matrix. Measurements were carried out in the spectral range of 4000–1000 cm^{-1}, with a spectral resolution of 4 cm^{-1} and 32 scans per pixel. The spatial resolution corresponded to a pixel size of approximately 5.5 µm. A defined central area of each filter was analyzed to ensure comparability between samples. The number of detected particles was corrected based on the ratio between the analyzed area and the total filter area. Spectral data were automatically pre-processed prior to polymer identification and subsequently analyzed using Purency Microplastics Finder software version 4.17. Polymer identification was based on comparison with reference spectral libraries integrated in the software and published polymer reference databases.

The analysis included the following steps:

- import of µFTIR chemical imaging data;
- automatic particle detection and segmentation;
- extraction of spectra for individual particles;
- comparison with reference spectral libraries;
- assignment of polymer type based on spectral similarity.

A match threshold of ≥70% was applied for reliable polymer identification. Only particles meeting this criterion were considered confirmed microplastics. The µFTIR imaging process, particle detection and spectral matching procedure are illustrated in Figure 5.

Microscopic inspection of filters

Microscopic observation of the filters was performed using a BSCOPE BS.1153-EPLH microscope (Euromex, Netherlands) equipped with a digital camera. Images were acquired at 40× magnification under reflected light conditions. The purpose of microscopic inspection was to document the morphology, size and shape of particles retained on the filter surface and to provide visual support for the µFTIR analysis. Microscopy was used as a complementary technique to verify particle presence and assist in the interpretation of µFTIR results.

Data analysis

Statistical processing of the data was performed using Microsoft Excel software. Microplastic abundance was expressed as the number of particles per kilogram of dry sediment (MPs kg^{-1}). The values were calculated by normalizing the number of detected particles to the mass of dry sediment used for extraction (50 g per sample). Since µFTIR analysis covered only a defined central area of each filter, the number of detected particles was corrected using a factor based on the

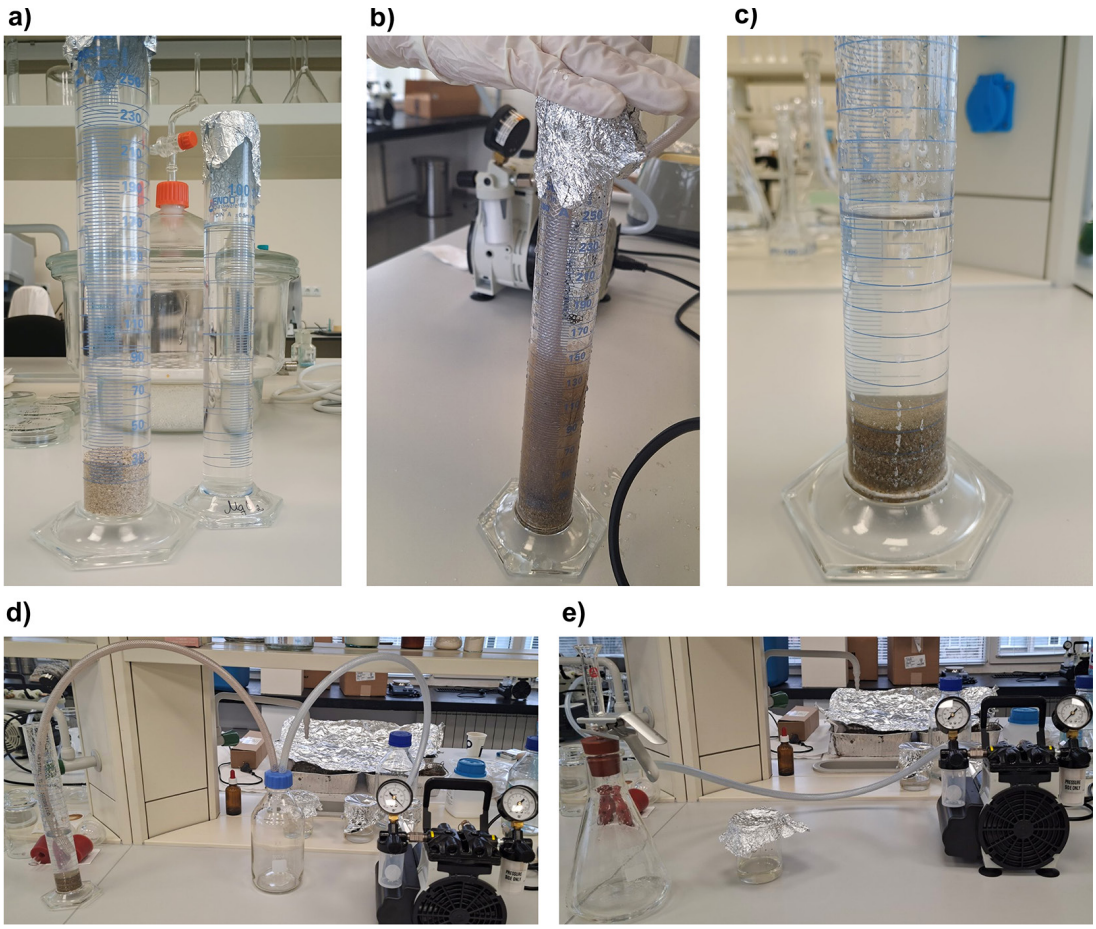

Figure 4. Laboratory procedures for microplastic extraction from sediments: (a) sediment mixed with flotation solution in a graduated cylinder; (b) bubbling system for particle separation; (c) sedimentation process; (d) decantation and collection of the supernatant after sedimentation; (e) vacuum filtration setup

ratio between the analyzed area and the total filter area. This correction allowed extrapolation of particle counts to the entire filter surface. Polymer composition was expressed as the relative proportion (%) of each polymer type with respect to the total number of identified microplastic particles. The results obtained for the two flotation solutions (NaCl and $MgCl_2$) were compared to evaluate differences in extraction efficiency. Due to the absence of biological and technical replicates, the results are presented descriptively and no inferential statistical tests were applied.

Quality control and contamination prevention

A number of precautions were taken to minimize the risk of external contamination with microplastic particles during laboratory processing. All laboratory vessels and instruments were pre-rinsed with distilled water and covered when not in use. Lab coats and gloves were used during the analysis to reduce the risk of contamination with synthetic fibers. The filtration systems and solutions used were pre-filtered to remove possible microplastic particles. Work surfaces were regularly cleaned and samples were processed in a controlled laboratory environment. To assess possible laboratory contamination, procedural blanks consisting of ultrapure water processed through the same extraction procedure were included. In addition, blank filters were exposed to laboratory air during sample preparation to evaluate potential airborne contamination. No microplastic particles morphologically consistent with the identified polymers were detected in the procedural blanks or airborne contamination controls. These results indicate that external contamination during sample preparation and analysis was negligible. Representative images of the blank filters and procedural controls are provided in the Supplementary Material.

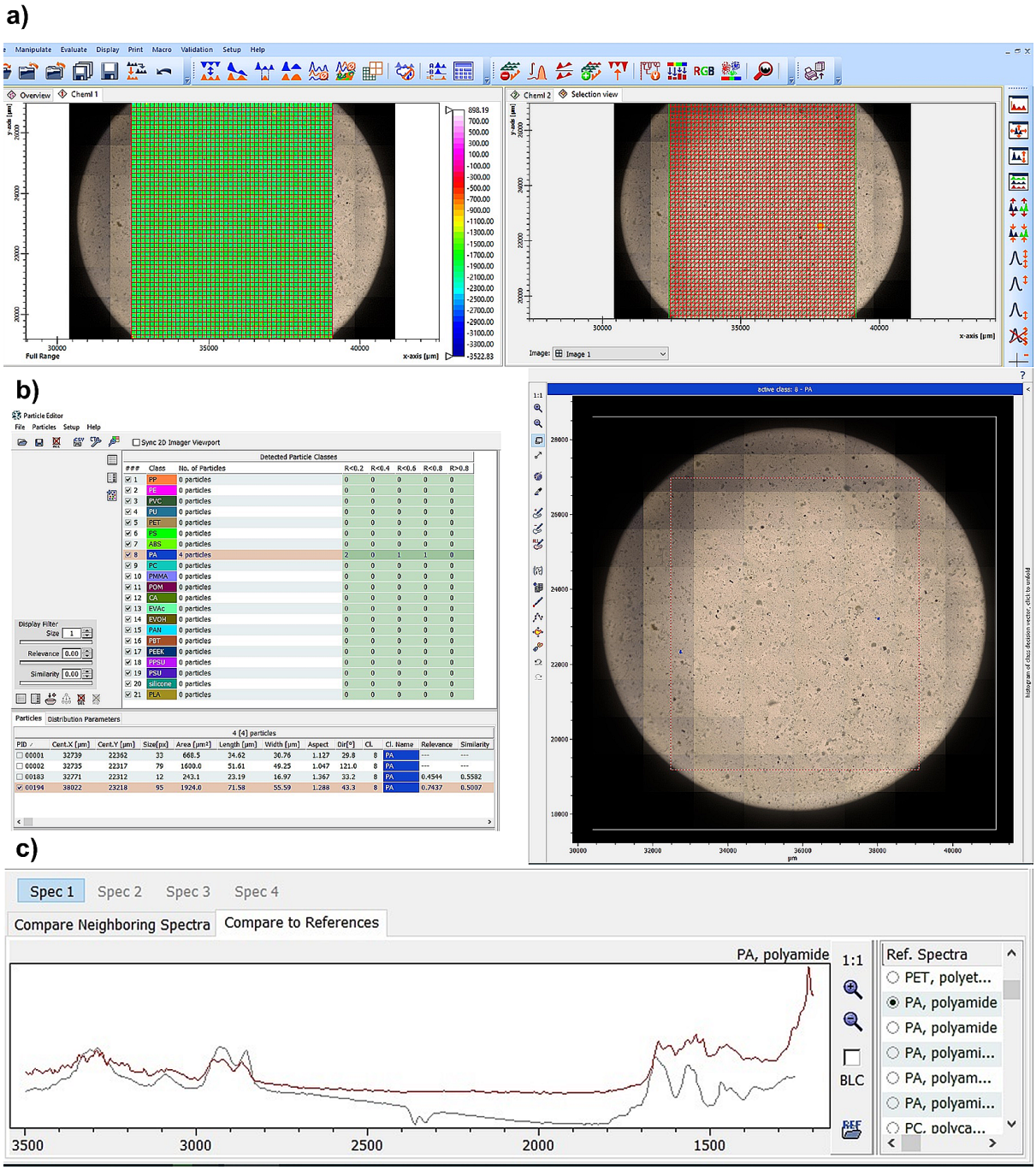

Figure 5. µFTIR analysis and data processing workflow: (a) chemical imaging of the filter surface; (b) automatic particle detection and segmentation using Purency Microplastics Finder software; (c) representative FTIR spectrum and comparison with reference libraries for polymer identification

Data availability

The datasets generated and analyzed during this study, including µFTIR spectra, particle counts and processed data, are available from the corresponding author upon reasonable request.

RESULTS AND DISCUSSION

Microplastic abundance and spatial distribution in sediments

Marine sediments are considered an important component of the marine environment, in which microplastics can accumulate as a result

of sedimentation processes and interaction with suspended particles and organic matter (Van Cauwenberghe et al., 2015; Woodall et al., 2014).

Microplastic particles were detected in the majority of the marine sediment samples collected from six stations in the Burgas Bay. The abundance of microplastics varied between the sampling stations and between the two flotation solutions used (NaCl and $MgCl_2$). In general, $MgCl_2$ yielded higher particle counts than NaCl, although higher values were observed with NaCl at stations S3 and S5. This may be related to the higher density of $MgCl_2$ (Cutroneo et al., 2021), which can improve the separation of denser particles from the sediment matrix (Imhof et al., 2012; Claessens et al., 2013). However, the relative performance of the two flotation solutions may also depend on sediment properties and the polymer composition of the recovered particles.

The detected values of microplastics ranged between 0.0 and 5497.8 MPs kg^{-1} dry sediment depending on the station and the flotation solution used (Table 2), indicating the presence of microplastics in the sediments of the studied water area of Burgas Bay. The highest values were found at station S5 near the island of St. Anastasia (5497.8 MPs kg^{-1} at NaCl), as well as at station S3 in the area of Chengene Skele (4712.4 MPs kg^{-1} at NaCl). The lowest value was recorded at station S6 during extraction with NaCl, where no confirmed microplastic particles were detected in the analyzed filter area. However, particles were identified in the corresponding $MgCl_2$ extract from the same station, indicating that this result should be interpreted cautiously and may reflect the lower recovery efficiency of NaCl for this specific sample rather than the complete absence of microplastics. Because particle abundances were extrapolated from a defined analyzed filter area to the total filter area, the zero value observed for station S6 should also be interpreted with caution. Representative microscopy images and μFTIR data for station S6 are provided in the Supplementary Material.

Spatial differences in the distribution of microplastics are observed between the studied areas of Burgas Bay, but no clear unidirectional spatial gradient is established. In general, the distribution of microplastics in the studied water area can be described as spatially heterogeneous, with higher values being recorded both in areas with more intense anthropogenic impact and in individual stations where local accumulation of particles is likely observed.

For a clearer assessment of spatial differences, the stations were grouped into three areas: Burgas Beach, Chengene Skele and St. Anastasia Island. Differences in the average microplastic abundance between the individual areas were observed. When extracting with NaCl, the average values ranged between 1374.5 MPs kg^{-1} in the Burgas Beach area and 2748.9 MPs kg^{-1} in the Chengene Skele and St. Anastasia Island areas. When using $MgCl_2$, the highest average value was found in the St. Anastasia Island area (3338.0 MPs kg^{-1}), followed by the Burgas Beach area (2552.6 MPs kg^{-1}), while the lowest values were recorded at Chengene Skele (1374.5 MPs kg^{-1}).

The distribution of microplastics between the individual stations is presented in Figure 6. When using NaCl solution, microplastic abundance varied between 0.0 MPs kg^{-1} at station S6 and 5497.8 MPs kg^{-1} at station S5, while when extracting with $MgCl_2$ the values varied between 1178.1 and 4712.4 MPs kg^{-1}. Relatively high microplastic abundance was also found at station S3 in the Chengene Skele area, where the value reached 4712.4 MPs kg^{-1} when extracted with NaCl. These results indicate considerable variability in the abundance of microplastics between the individual stations, which suggests the influence of local factors such as anthropogenic activity, particle transport by sea currents and sedimentation processes in the coastal zone. Higher values of microplastics were found at some stations located near coastal areas with more intensive human activity. Similar results have been reported in other studies, which show that coastal areas located near urban areas and port infrastructure are often characterized by higher concentrations of microplastics in sediments (Claessens et al., 2013; Van Cauwenberghe et al., 2015). Burgas Bay is a semi-closed coastal system in which hydrodynamic processes and particle transport can contribute to the local accumulation of microplastics in sediments. The detected microplastic values are comparable to concentrations reported in other studies of microplastics in coastal marine sediments (Cincinelli et al., 2021).

In addition to local sources of pollution, the distribution of microplastics in sediments can also be influenced by hydrodynamic processes such as currents, waves and sediment resuspension, which control the transport and accumulation of particles in coastal areas. Similar spatial variations have been observed in other coastal marine areas, where urban areas and port areas have been

Table 2. Abundance of microplastics in sediment samples expressed as MPs kg^{-1} dry sediment

Site	NaCl raw count	$MgCl_2$ raw count	NaCl corrected count	$MgCl_2$ corrected count	NaCl MPs kg^{-1}	$MgCl_2$ MPs kg^{-1}	Area
S1	4	6	78.5	117.8	1570.8	2356.2	Burgas Beach
S2	3	7	58.9	137.4	1178.1	2748.9	Burgas Beach
S3	12	3	235.6	58.9	4712.4	1178.1	Chengene Skele
S4	2	4	39.3	78.5	785.4	1570.8	Chengene Skele
S5	14	12	274.9	235.6	5497.8	4712.4	St. Anastasia Island
S6	0	5	0.0	98.2	0.0	1963.5	St. Anastasia Island

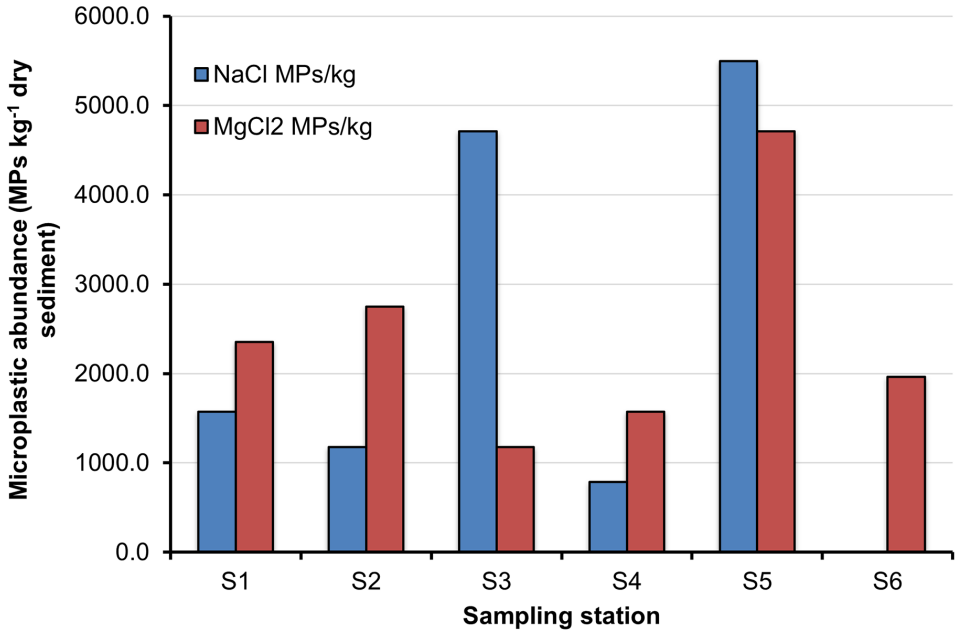

Figure 6. Microplastic abundance in sediment samples collected from six stations in Burgas Bay, expressed as MPs kg^{-1} dry sediment. Bars represent the abundance of microplastic particles obtained after density separation using two flotation solutions (NaCl and $MgCl_2$)

identified as potential "hotspots" for the accumulation of microplastics (Hidalgo-Ruz et al., 2012; Van Cauwenberghe et al., 2013). The determined values of microplastics in the sediments of Burgas Bay are comparable to concentrations reported in other studies of coastal marine sediments (Van Cauwenberghe et al., 2013), indicating that microplastic pollution is widespread in coastal marine ecosystems. Because no biological or technical replicates were included, the observed differences between stations and flotation solutions should be interpreted descriptively rather than statistically.

Comparison of the effectiveness of the flotation solutions used

The comparison of the average microplastic abundance obtained with the two flotation solutions used is presented in Figure 7. $MgCl_2$ yielded higher particle counts at four of the six stations, whereas NaCl yielded higher values at stations S3 and S5. This station-specific variability suggests that the relative performance of the two flotation solutions may depend not only on solution density, but also on sediment properties and the polymer composition of the recovered particles.

The somewhat higher average abundance obtained with $MgCl_2$ may be related to the higher density of this solution, which may improve the separation of denser microplastic particles from the sediment matrix. Although saturated NaCl solution is often used due to its low cost and relative safety (Claessens et al., 2013), its density is not always sufficient to extract denser polymers such as PVC or PET (Hidalgo-Ruz et al., 2012). Similar results have been reported in other studies, which show that higher density solutions

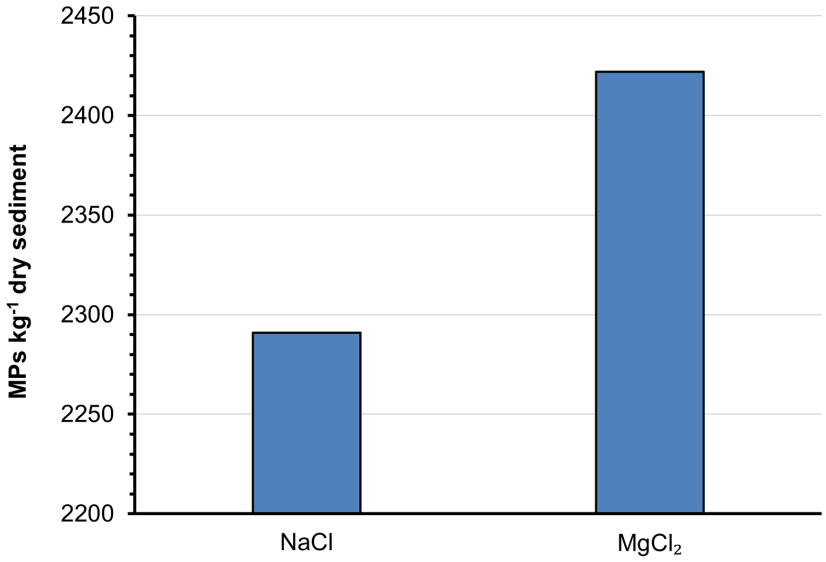

Figure 7. Mean microplastic abundance extracted from sediment samples using two density separation solutions (NaCl and $MgCl_2$), expressed as MPs kg^{-1} dry sediment

can improve the recovery of microplastics from sediment samples (Imhof et al., 2012; Claessens et al., 2013). However, the results show that the efficiency of flotation solutions varies between stations, which is likely due to differences in the polymer composition, density and size distribution of microplastic particles, as well as the specific characteristics of the sediment matrix. Overall, the average microplastic abundance extracted with $MgCl_2$ (≈2422 MPs kg^{-1}) is slightly higher than that obtained with NaCl (≈2291 MPs kg^{-1}).

Because no biological or technical replicates were included, the observed differences between flotation solutions and sampling stations should be interpreted descriptively. Additional replicated sampling and repeated extractions are needed to statistically confirm these trends.

Polymer composition of microplastic particles

The polymer composition of the identified microplastic particles was determined by μFTIR analysis. The obtained spectra were compared with reference spectral libraries, which allowed a reliable determination of the polymer type.

The μFTIR analysis allowed the identification of nine different polymer types in the sediment samples from Burgas Bay (Table 3). A total of 72 microplastic particles were identified by μFTIR analysis. Although the total number of identified particles was relatively low, the dataset provides initial polymer-specific information for marine sediments from Burgas Bay. Therefore, the polymer composition results should be interpreted as indicative rather than fully representative. The largest relative share is held by polyamide (PA) particles, which represent 48.6% of all identified microplastics, followed by polypropylene (PP) – 15.3%, as well as ethylene vinyl alcohol (EVOH) and polyethylene (PE), which represent 11.1% each. The remaining polymers, including PVC, PMMA, PS, ABS and PET, were detected in smaller quantities. The percentage distribution of the detected polymers is presented in Figure 8. Polyamide and polypropylene together represent approximately 64% of all identified microplastics, which suggests a significant contribution of fishing activities to microplastic pollution in the studied water area.

The predominance of polyamide particles suggests a possible link to fishing activities in the area and is probably related to the use of this polymer in the manufacture of fishing gear such as nets, ropes and other fishing gear. Similar results have been reported in other studies of the marine environment, where polyamide fibers are often associated with fishing activities and maritime transport (Andrady, 2011; Hidalgo-Ruz et al., 2012). The detected presence of PS is likely the result of the fragmentation of packaging materials and expanded polystyrene products, widely used in coastal urban areas (Cole et al., 2011). Polyvinyl chloride (PVC) is also a widely used industrial polymer that can enter the marine environment through various anthropogenic sources,

Table 3. Polymer types identified in microplastic particles from sediment samples collected in Burgas Bay determined by μFTIR analysis

Polymer	Total count	NaCl count	$MgCl_2$ count	Percentage (%)
ABS	1	1	0	1.4
EVOH	8	8	0	11.1
PA	35	14	21	48.6
PE	8	6	2	11.1
PET	1	0	1	1.4
PMMA	2	1	1	2.8
PP	11	4	7	15.3
PS	2	0	2	2.8
PVC	4	1	3	5.6
Total	72	35	37	100.0

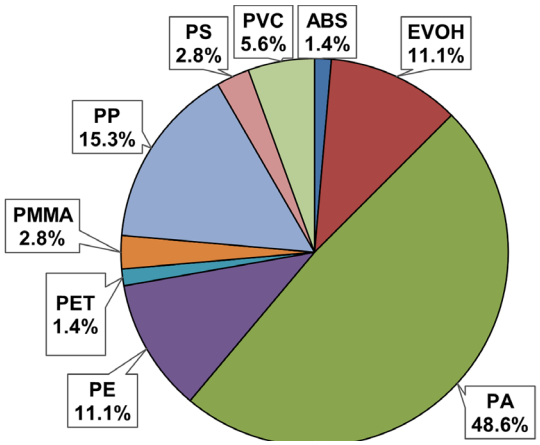

Figure 8. Polymer composition of microplastic particles identified in sediment samples using μFTIR analysis

including construction materials from port infrastructure and industrial waste (Andrady, 2011).

A similar polymer composition has been reported in other studies of microplastics in the Black Sea, where polyolefin polymers such as polyethylene and polypropylene, associated with fishing activities, coastal urbanization and maritime transport, dominate (Aytan et al., 2016; Cincinelli et al., 2021). The results obtained show that local anthropogenic activities likely play a significant role in the formation of microplastic pollution in the sediments of Burgas Bay.

The distribution of polymer types between the individual stations is presented in Figure 9. The obtained results show a clear dominance of polyamide (PA) among the identified microplastic particles in most of the studied stations. The largest number of particles was found at station S5, where the greatest diversity of polymer types was also observed. However, these station-specific differences should be interpreted cautiously because of the limited number of identified particles and the absence of replicate analyses. In addition to PA, PP, PE, EVOH, as well as smaller amounts of PVC, PMMA, PET, PS and ABS were also identified in the samples. The obtained distribution shows variability between the individual stations, which suggests the influence of both local pollution sources and processes of transport and accumulation of microplastics in the coastal zone of the Burgas Bay.

To confirm the polymer composition, microplastic particles were analyzed by μFTIR spectroscopy. Figure 10 presents optical microscopy images and μFTIR characterization of the identified particles. Panels (a–d) show optical microscopy images of four representative microplastic particles, illustrating different morphologies. Panel (e) presents the spatial distribution of the identified polymer types (PA, PE, PP, PVC) in the analyzed samples. Panels (f) and (g) show representative μFTIR spectra of particles identified as PA and PS, respectively. Optical imaging and μFTIR analysis enabled accurate localization and identification of microplastic particles on the filter surface. The observed spectral features show good agreement with reference spectra for the corresponding polymers. The results support the reliability of the μFTIR method used for identification of microplastic particles and are consistent with the spectral characteristics described in other studies of microplastics in the marine environment (Hidalgo-Ruz et al., 2012; Primpke et al., 2018). Because only 72 particles were identified in total, the polymer composition and spatial

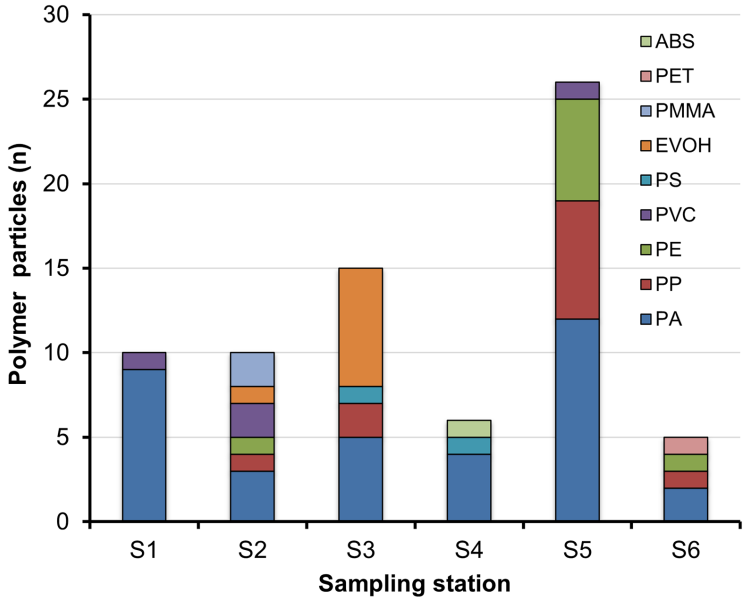

Figure 9. Distribution of polymer types identified in microplastic particles at the six sampling stations in Burgas Bay based on μFTIR analysis. Polymer counts represent the total number of particles detected in samples extracted using both density separation solutions (NaCl and $MgCl_2$)

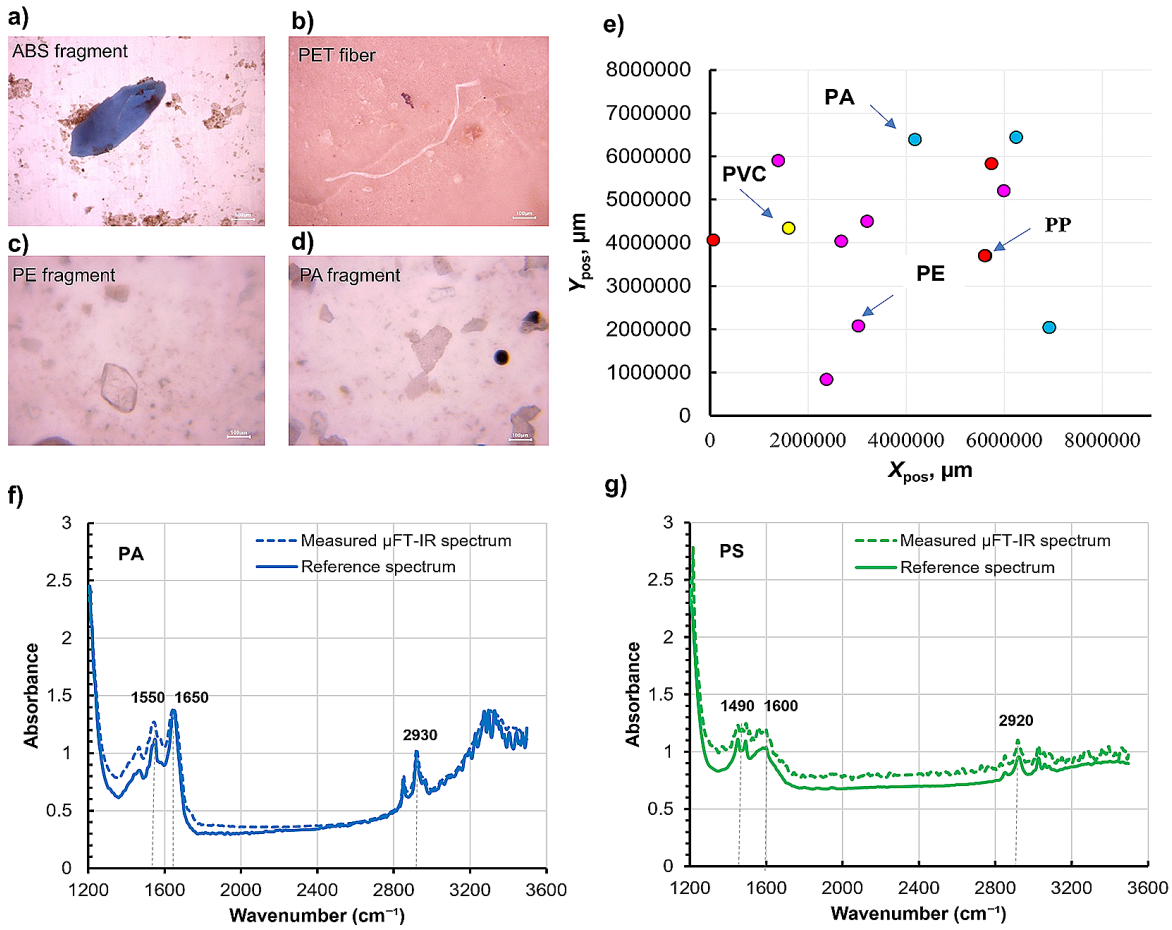

Figure 10. Representative optical microscopy images and μFTIR characterization of microplastic particles detected in sediment samples from Burgas Bay (Black Sea): (a–d) optical microscopy images of representative microplastic particles; (e) spatial distribution of identified polymer types (PA, PE, PP, PVC) in sediment samples; (f) μFTIR spectrum of a particle identified as PA; (g) μFTIR spectrum of a particle identified as PS

patterns presented here should be considered preliminary and indicative for the investigated sites.

Size distribution of microplastic particles

The morphological characteristics of the identified microplastic particles were assessed by analyzing their geometric parameters, including length, width and aspect ratio, obtained by μFTIR imaging analysis. The length/width ratio of the particles varies over a wide range, with most particles having aspect ratios close to 1–2, indicating that a large proportion of the particles are characterized by a relatively irregular shape, typical of fragmented plastic materials. The analysis shows that the size of microplastic particles varies over a wide range, with the measured particle length between 4.5 and 1600.3 μm. The largest particle was found at station S4 and was identified as ABS. The majority of the identified particles were below 100 μm in size, indicating a predominance of relatively small microplastic particles in the studied sediments.

The size distribution of microplastic particles is presented in Figure 11, with the largest number of particles found in the smaller size classes (0–25 μm and 25–50 μm). This distribution suggests that a significant proportion of the particles likely originate from secondary fragmentation of larger plastic materials in the marine environment. The detection of a large number of small particles can also be explained by the use of μFTIR imaging analysis, which allows the detection and identification of microplastic particles down to approximately 5 μm in size, which significantly improves the ability to register finer particles compared to traditional visual methods.

The presence of a large number of small particles is of particular importance, as they can more easily interact with organisms in the marine environment and participate in the processes of transport and accumulation of pollutants. Similar observations of the dominance of small microplastic particles in the marine environment have been reported in other studies (Hidalgo-Ruz et al., 2012; Andrady, 2011). The Black Sea is a semi-enclosed sea basin with limited water exchange with other sea basins, which favors the accumulation of various pollutants, including microplastics. Similar results have been reported in other studies, which show a wide distribution of microplastics in different components of the marine environment in the Black Sea (Aytan et al., 2016; Cincinelli et al., 2021).

The present study has several limitations that should be taken into account. Sediment samples were collected within a single season and no biological or technical replicates were included, which restricts statistical assessment of variability between stations and flotation solutions. In addition, μFTIR analysis was performed on a defined central area of each filter, and the number of particles was corrected to the total filter area.

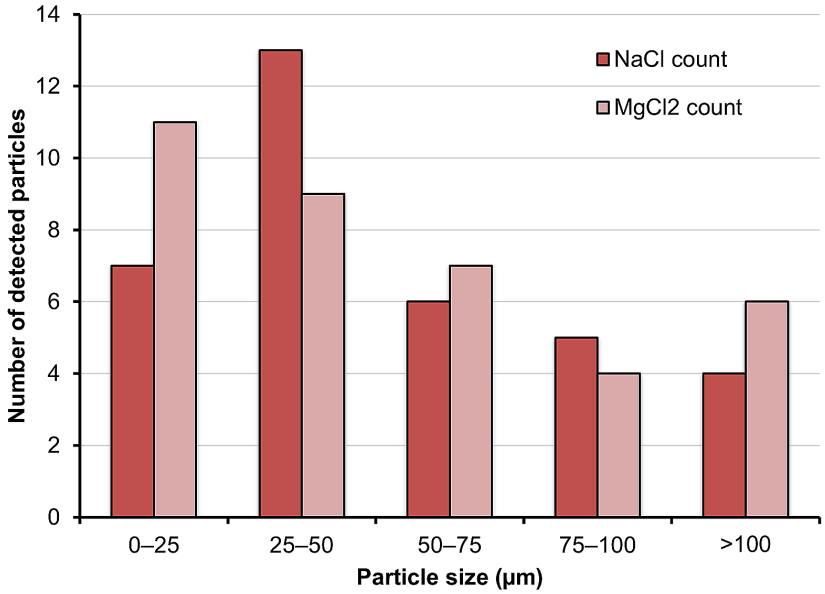

Figure 11. Size distribution of microplastic particles identified in sediment samples from Burgas Bay based on μFTIR measurements of particle length. The distribution is shown for particles extracted using NaCl and $MgCl_2$ flotation solutions

Because particle distribution on the filter surface may be heterogeneous, this extrapolation introduces additional uncertainty that could not be quantified in the present study. A further limitation is the relatively small number of confirmed microplastic particles (n = 72), which restricts broader ecological interpretation. Despite these limitations, the results provide initial baseline data on microplastic occurrence and polymer composition in marine sediments from Burgas Bay and may serve as a basis for future studies incorporating replicated sampling, repeated extractions, seasonal monitoring and improved quantification approaches.

CONCLUSIONS

Microplastics were detected in marine sediments from Burgas Bay, with abundances ranging from 0 to 5497.8 MPs kg^{-1} dry sediment, confirming the presence of plastic contamination in this intensively used coastal area. The observed spatial variability suggests heterogeneous distribution patterns; however, these findings should be interpreted with caution due to the limited replication, which constrains robust statistical inference. The comparative analysis of density separation solutions indicates that $MgCl_2$ generally enhanced the recovery of microplastic particles relative to NaCl, supporting the hypothesis that higher-density solutions improve extraction efficiency. Nevertheless, the lack of consistency across all sampling stations highlights that recovery efficiency is not solely controlled by solution density but is also influenced by sediment characteristics and particle properties. μFTIR imaging enabled the identification of nine polymer types, with polyamide, polypropylene, and polyethylene being the most frequently detected. However, the relatively low number of identified particles limits the representativeness of the observed polymer distribution and underscores the importance of increasing analytical throughput in future studies.

Overall, this study provides new methodological and baseline environmental data on microplastics in Burgas Bay and the Bulgarian coastal sector of the Black Sea. The findings demonstrate that the choice of density separation solution can influence both the abundance and polymer composition of microplastics, thereby affecting the comparability of results across studies.

Acknowledgments

This research was funded by the Bulgarian National Science Fund under the contract KP-06-H89/4 “Investigation of the content of micropolymers in sediment and biota from a marine ecosystem”.

REFERENCES

1. Andrady, A. L. (2011). Microplastics in the marine environment. *Marine Pollution Bulletin*, *62*(8), 1596–1605. https://doi.org/10.1016/j.marpolbul.2011.05.030
2. Aytan, U., Valente, A., Senturk, Y., Usta, R., Sahin, F. B. E., Mazlum, R. E., Agirbas, E. (2016). First evaluation of neustonic microplastics in Black Sea waters. *Marine Environmental Research*, *119*, 22–30. https://doi.org/10.1016/j.marenvres.2016.05.009
3. Berov, D., Klayn, S. (2020). Microplastics and floating litter pollution in Bulgarian Black Sea coastal waters. *Marine Pollution Bulletin*, 153, 110964. https://doi.org/10.1016/j.marpolbul.2020.110964
4. Bobchev, N., Dimitrov, M., Todorova, V. (2024). High microplastic pollution in marine sediments associated with urbanised coastal areas of the southwestern Black Sea. *Marine Pollution Bulletin*, *198*, 115980. https://doi.org/10.1016/j.marpolbul.2023.115980
5. Cincinelli, A., Scopetani, C., Chelazzi, D., Lombardini, E., Martellini, T., Pogojeva, M., Slobodnik, J. (2021). Microplastics in the Black Sea sediments. *Science of the Total Environment*, 760, 143898. https://doi.org/10.1016/j.scitotenv.2020.143898
6. Claessens, M., Van Cauwenberghe, L., Vandegehuchte, M. B., Janssen, C. R. (2013). New techniques for the detection of microplastics in sediments and field collected organisms. *Marine Pollution Bulletin*, 70(1–2), 227–233. https://doi.org/10.1016/j.marpolbul.2013.03.009
7. Cole, M., Lindeque, P., Halsband, C., Galloway, T. S. (2011). Microplastics as contaminants in the marine environment: A review. *Marine Pollution Bulletin*, *62*(12), 2588–2597. https://doi.org/10.1016/j.marpolbul.2011.09.025
8. Cutroneo, L., Reboa, A., Geneselli, I., Capello, M. (2021). Considerations on salts used for density separation in the extraction of microplastics from sediments. *Marine Pollution Bulletin*, *166,* 112216. https://doi.org/10.1016/j.marpolbul.2021.112216
9. Hidalgo-Ruz, V., Gutow, L., Thompson, R. C., Thiel, M. (2012). Microplastics in the marine environment: A review of the methods used for identification and quantification. *Environmental Science & Technology*, *46*(6), 3060–3075. https://doi.

org/10.1021/es2031505

10. Holmes, L. A., Turner, A., Thompson, R. C. (2012). Adsorption of trace metals to plastic resin pellets in the marine environment. *Environmental Pollution*, *160*, 42–48. https://doi.org/10.1016/j.envpol.2011.08.052
11. Imhof, H. K., Schmid, J., Niessner, R., Ivleva, N. P., Laforsch, C. (2012). A novel, highly efficient method for the separation and quantification of plastic particles in sediments. *Limnology and Oceanography: Methods*, *10*, 524–537. https://doi.org/10.4319/lom.2012.10.524
12. Löder, M. G. J., Gerdts, G. (2015). *Methodology used for the detection and identification of microplastics*. In M. Bergmann, L. Gutow, M. Klages (Eds.), Marine anthropogenic litter (pp. 201–227). Springer. https://doi.org/10.1007/978-3-319-16510-3_8
13. Nuelle, M. T., Dekiff, J. H., Remy, D., Fries, E. (2014). A new analytical approach for monitoring microplastics in marine sediments. *Environmental Pollution*, *184*, 161–169. https://doi.org/10.1016/j.envpol.2013.07.027
14. Prata, J. C., da Costa, J. P., Duarte, A. C., Rocha-Santos, T. (2019). Methods for sampling and detection of microplastics in water and sediment: A critical review. *TrAC Trends in Analytical Chemistry*, *110*, 150–159. https://doi.org/10.1016/j.trac.2018.10.029
15. Primpke, S., Lorenz, C., Rascher-Friesenhausen, R., Gerdts, G. (2017). An automated approach for microplastics analysis using focal plane array (FPA) FTIR microscopy and image analysis. *Analytical Methods*, *9*(9), 1499–1511. https://doi.org/10.1039/c6ay02476a
16. Primpke, S., Wirth, M., Lorenz, C., Gerdts, G. (2018). Reference database design for the automated analysis of microplastic samples based on FTIR spectroscopy. *Analytical and Bioanalytical Chemistry*, *410*, 5131–5141. https://doi.org/10.1007/s00216-018-1156-x
17. Rochman, C. M., Hoh, E., Hentschel, B. T., Kaye, S. (2013). Ingested plastic transfers hazardous chemicals to fish and induces hepatic stress. *Scientific Reports*, *3*, 3263. https://doi.org/10.1038/srep03263
18. Shim, W. J., Hong, S. H., Eo, S. (2018). *Marine microplastics: Abundance, distribution, and composition.* In E. Y. Zeng (Ed.), Microplastic contamination in aquatic environments (pp. 1–26). Elsevier. https://doi.org/10.1016/B978-0-12-813747-5.00001-1
19. Turmanova, S. H., Hristov, Y. H., Kiryakova, D. S., Ivanova, E. D., Atanasova, P. V., Kolchakova, G. R., Ilieva, A. S., Mollova, E. Y., Dimitrov, A. N., Todorov, N. S., Grigorova, G. G. (in press). Extraction of microplastics from marine sediment in Burgas Bay, Black Sea. *Bulgarian Chemical Communications*.
20. Van Cauwenberghe, L., Vanreusel, A., Mees, J., Janssen, C. R. (2013). Microplastic pollution in deep-sea sediments. *Environmental Pollution*, *182*, 495–499. https://doi.org/10.1016/j.envpol.2013.08.013
21. Van Cauwenberghe, L., Vanreusel, A., Mees, J., Janssen, C. R. (2015). Microplastics in sediments: A review of techniques, occurrence and effects. *Marine Environmental Research, 111*, 5–17. https://doi.org/10.1016/j.marenvres.2015.06.007
22. Woodall, L. C., Sanchez-Vidal, A., Canals, M., Paterson, G. L. J., Coppock, R., Sleight, V., Calafat, A., Rogers, A. D., Narayanaswamy, B. E., Thompson, R. C. (2014). The deep sea is a major sink for microplastic debris. *Royal Society Open Science*, *1*(4), 140317. https://doi.org/10.1098/rsos.140317
23. Wright, S. L., Thompson, R. C., Galloway, T. S. (2013). The physical impacts of microplastics on marine organisms. *Environmental Pollution*, *178*, 483–492. https://doi.org/10.1016/j.envpol.2013.02.031