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*Article*

# Preliminary study on FTIR analysis of microplastics in water samples of Erzeni River mouth, Adriatic Sea—Albania

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***Abstract***

*There is a significant gap in the published literature regarding the characterization and analysis of microplastics (MPs) in the aquatic ecosystems of Albania. This study focuses on the delta of the Erzeni River as one of the major sources of anthropogenic pollution, including micro- and macroplastics, in the Albanian coastal area of the Adriatc Sea. After oxidative treatment of water samples with $H_2O_2$ and separation using the high-density method, identification of MPs was performed using spectroscopic and FTIR methods. Spectroscopic and FTIR analysis revealed the presence of MPs of the type PVC or PVC/MMT, nylon, PS, and PE in water samples. Spectral bands in the region of 400 $cm^{-1}$ to 1100 $cm^{-1}$ can be associated with the presence of synthetic rubber. The analysis presented in this study investigates the potential sources of MPs in coastal environments, based on the identification of plastic species. This research is the first of its kind in Albania's coastal area that serves as an important initial contribution to the development of a database dedicated to the assessment of MPs in this ecosystem. MPs in water samples from the delta of the Erzeni River that discharges into the Adriatic Sea. The objective of this study was to examine the morphological characteristics and classifications of MPs found in the research area, as well as to assess the consequences of their degradation.*

***Keywords:*** *microplastics, Erzeni River Delta, microscopic images, FTIR, Adriatic Sea, Albania*

## INTRODUCTION

Extensive utilization of plastic materials in industry, agriculture, and personal use has resulted in an increase in their production and, consequently, in an increase in the generation of plastic waste. However, a prominent issue related to this trend is the management of waste generated during the production phase, as well as after the product life cycle [1, 2]. The problem of plastic waste is related to the lack of adequate implementation of disposal methods and the low recycling rate. The evolution of plastic waste management from 2006 to 2020 has highlighted the interaction between landfilling, energy recovery, and recycling [3, 4] also attribute the accumulation of plastic in terrestrial and marine ecosystems to the lack of public awareness.

Over the past decades, the accumulation of microplastic waste in the environment has created a serious ecological issue. Plastic pollution has become a crucial global problem, affecting the environment and human health and posing a high threat to aquatic flora and fauna and indirectly to humans as consumers of aquatic products [5, 6].

The presence of MPs in various ecosystems is considered highly dangerous when exist in the form of very small particles [6]. MPs refer to small solid particles with dimensions smaller than 5 mm made of non-biodegradable polymers such as polyethylene (PE), polyester (PES), polyvinyl chloride

(PVC), and high-density polyethylene (HDPE) [4, 7÷10]. Fragments, foams, granules, fibbers, and films are the most prevalent types of MPs in the environment. MPs in the environment primarily exist as fragments, foams, granules, fibbres, and films. They can originate from several industrial processes [8, 10], from natural breakdown due to ultraviolet light exposure, and from various physical, chemical, and microbial interactions [6, 8, 11]. The problem of plastic pollution in coastal zones has gained considerable attention from researchers. Studies show that coastal areas are particularly vulnerable and are heavily affected by contamination caused by plastic waste. The growing rates of illness in marine organisms, along with a decline in fishing yields, have immediate consequences for the food web, primarily due to the accumulation and concentration of MPs within aquatic species. Albania produces approximately 1.08 million tons of plastic waste annually, with a large portion ultimately disposed of in landfills. Around 18% of this waste is recycled, a significant amount is incinerated in open fields, and about 0.8% enters the ocean via the river discharges [5].

The study of MPs in various ecosystems is still in its early stages and has not yet progressed to the standardized analytical methodologies and the establishment of thresholds to safeguard human health and biodiversity. In the absence of a standardized methodology, various approaches for sampling, sample processing, chemical identification, and size characterization have been utilized to monitor MPs in aquatic ecosystems [2, 6, 12, 13]. Prata et al. [14] recommend 1 liter of water sample [15] collected by bulk sampling [13] is considered adequate for a rough assessment of MPs concentrations in water sample contain organic and mineral elements. This method only captures the environmental conditions at the specific location and time of sampling [13]. Nevertheless, the slight fluctuations in continuous monitoring of yearly and monthly physical-chemical parameters in coastal regions demonstrate the value of implementing discrete monthly or seasonal monitoring programs with specific measurements of these parameters [16].

Besides, the abundance of organic matter solid materials in samples is a considerable problem when waters from surface sources are examined. Density separation served as the initial step for isolating MPs from contaminants within the sample matrix. This technique is based on the differences in densities of MPs, which range from 0.91 g/cm³ for polypropylene (PP) to 1.45 g/cm³ for polyvinyl chloride (PVC), and water samples [6, 17÷19]. To achieve a good separation, the density of the water samples could be modified by adding various electrolytes such as NaCl, $ZnCl_2$, NaI, or $CaCl_2$ [13, 14, 18]. NaCl is often utilized as a safe chemical option, primarily because of the simplicity of preparing the aqueous solution. Following the addition of NaCl, the density of the samples was adjusted to 1.7 g/cm³. in addition, different digestion techniques, such as oxidant digestion, acid digestion, alkaline digestion, and enzymatic digestion, are utilized for purification of the water matrix [6, 18, 20, 21]. The water samples with high TSS and organic matter content were treated with density separation and oxidative digestion by hydrogen peroxide. Shi et al. [22] proposed filtration of the sample followed by oxidative dissolution of the organic matter in water using $H_2O_2$ 30%.

Finally, MPs measuring can be detected using stereomicroscopic techniques. Enlarged microscopic images offer more detailed information about the surface structure and composition of the particles, which is crucial for understanding the topology of plastic particles present in the sample [9]. During microscopic investigation, color is an essential parameter for the possible identification of plastic waste, as well as possible contamination during sample preparation [1, 23]. MPs have been reported in a range of colors, including red, orange, yellow, brown, tan, white, gray, blue, green, etc. [1, 23]. Blue and red fibers are frequently reported, while black, white, transparent, or translucent particles may be overlooked during visual investigation [1].

The goal of this research is to identify the impact of MPs on marine pollution in Albania's coastal regions and to tackle a significant environmental challenge linked to MP contamination in this area. The primary objective is to assess the pollution levels of water samples collected from the Erzeni River delta, which will assist in determining whether samples with elevated organic matter content or those that are cleaner necessitate chemical treatment prior to MP analysis. The next priority is to

optimize the analytical method, which includes sampling as well as physical and chemical treatments, leading to microscopic and spectroscopic analyses for the identification of MPS in water samples. By investigating this specific case study alongside empirical data and the most recent techniques for detecting MPS in water, the research aims to gain a comprehensive understanding of this intricate issue and to pinpoint the most effective strategies for safeguarding the environment and public health.

## MATERIALS AND METHODS

*Description of the study area*

The Albanian coastline along the Adriatic Sea stretches for about 284 kilometers. The growth of urban areas and an increase in the tourism industry bring about environmental and social transformations. The levels of water salinity, as well as the physical, chemical, and nutrient contents, are primarily influenced by the input of freshwater from river discharges, especially in the river deltas. The typical discharge of rivers in Albania ranges from 940 to 1,100 $m^3/s$, making these waterways a significant source of freshwater. In contrast, the primary tributary of the Adriatic Sea, the Po River, has an average discharge of 1,500 $m^3/s$ during the simulation period [24]. The average outflow data ($m^3/sec$) [24] and average sediment loads (MT/year) [25] of Albanian rivers indicated that five Albanian rivers discharge approximately 85 Mtyr–1 sediment. The average outflow of Erzeni River is 18.1 $m^3/sec$ [24] and average sediment loads of 4 MT/year [25].

*Sampling*

Water samples were collected from three points and mixed together by homogenization to create a representative sample. Sampling was done in October 2023. The position of sampling site is shown in Figure 1.

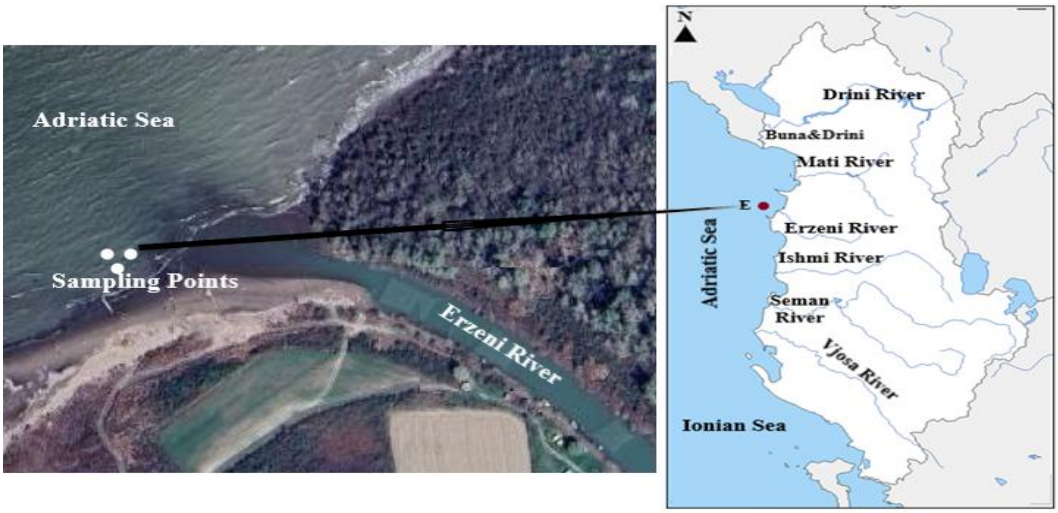


**Fig. 1**. The map of sampling site and corresponding geographic coordinates (N 41.436667; E 19.458957)

The sampling was carried out based on the recommendation presented by Prata et al. [25]. According to it, 1 litter of water packed in glass bottles was taken for analysis. The bottles were wrapped in aluminium foil and transported to the laboratory on the same day. In the lab, filtration with 5 mm sieve was applied before the analysis [2, 6, 12]. Water samples utilized for chemical analysis were collected in 1.5 L PET bottles cleaned in the laboratory with 1:1 $HNO_3$ and rinsed with deionized water. The water samples were maintained in refrigeration at -4ºC before the analysis.

*Pretreatment of the samples and identification methods*

Two types of pretreatments of surface water samples were applied: separation using density and chemical digestion.

For identification of the microplastics were used microscopic analysis and Fourier-transform infrared (FTIR) spectroscopy.

*Density separation*
To achieve a good separation of MPs, with density ranged from 0.91 g/cm³ for polypropylene (PP) to 1.45 g/cm³ for polyvinyl chloride (PVC), from the water, the density of the water sample was adjusted by adding NaCl at concentration 25 % and temperature 10ºC. Under these conditions, the lower-density polymers rise to the surface of the solution after being shaken and allowed to settle in a one-liter separation funnel. The solid-phase particles that settled at the bottom of the funnel were carefully removed.

*Chemical digestion*
The water samples were treated with 30% $H_2O_2$ (Hydrogen peroxide 30%, analytical grade, Merck, Germany) solution in a thermostatically controlled water bath at 40÷50ºC until the solution appeared clear. After cooling at room temperature, the water samples underwent vacuum filtration using Whatman GF/A glass microfiber filters (diameter 47 mm), designed for high–efficiency filtration process.

*Microscopic identification of MPs*
Microscopic analysis was performed at the Faculty of Natural Sciences, Department of Physics. The Kozo XJP304 microscope (Nanjing KOZO Optical and Electronical Instrument Co., LTD, China) was used to analyze MPs in water samples comes with the following characteristics: a magnification level of 100X, a halogen lamp as the light source (12V and 50W), and an Abbe condenser that has a numerical aperture of 1.25. Furthermore, it includes a digital camera, specifically the Sony TCC-8.1, and employs image analysis software referred to as View version 7.3.1.7. The characteristics of MPs, including their color, shape, and size, are used to classify the specific type of MPs. The classification based on color was performed following the polymer cluster map created by Helmholtz [23].

*FTIR analysis*
FTIR was utilized to identify the chemical composition of MPs present on filters obtained from the treatment of water samples. Measurements were performed using a Bruker Alpha II FTIR spectrometer (Bruker Corporation, Berlin, Germany) equipped with an attenuated total reflectance (ATR) accessory and a diamond crystal window. Spectra were acquired in absorption mode over the spectral range of 4000÷400 $cm^{-1}$, using 50 averaged scans. Depending on the MP content in the sample, a resolution of 2÷4 $cm^{-1}$ was applied. Measurements at the spots identified in the filter material by means of a microscope inspection were used to conduct the FTIR analysis of MPs. The spectra are registered after background correction in reference to the spectrum of a pure filter treated with blank reagents. The type and chemical composition of polymers were identified by comparing the FTIR spectra of the samples with the standard spectra of pure polymers available online [26÷31], as well as the characteristic bands published in the review papers of various authors [10, 22]. Manual interpretation of each absorption peak compared with the reference tables was used in the evaluation of MPs of current samples.

## RESULTS AND DISCUSSION

The experimental design for MPs identification must consider the variables of the sampling site that will likely influence the resultant data, including the potential proximity to the sources and the physical and chemical properties of the sample matrix [6]. Physical-chemical parameter analyses were implemented to assess the matrix of the water samples. The outcomes of these analyses are summarized in the Table 1.

**Table 1.** Physical-chemical parameters in water samples

| Parameter | Salinity | Alkalinity | TSS | Dissolved oxygen (DO) | Biological oxygen demand ($BOD_5$) | Chemical oxygen demand (COD) |
|---|---|---|---|---|---|---|
| Value (mg/L) | 0.15 | 269 | 96 | 6.50 | 5.00 | 8.00 |
| Parameter | $N\text{-}NH_4^+$ | $P\text{-}PO_4^{3-}$ | TOC | Conductivity | *Escherichia Coli* | *Escherichia Intestinal* |
| Value | 0.50 mg/L | 0.15 mg/L | 7 mg/L | 572 µS/cm | 2100 cfu/100 ml | 265 cfu/100 ml |

TSS - total suspended solids; TOC - total organic carbon

Elevated levels of BOD, COD, and ammonium exceeding the WHO guidelines [32] for surface water (2 mg/L, 10 mg/L, and 0.3 mg/L, respectively) indicate pollution state. A high concentration of ammonium in surface water suggests contamination from agricultural activities, sewage, and other sources of nitrogen pollution. Water samples revealed moderate content of chemical parameters (BOD, COD, and TOC), nutrients ($NH_4^+$ and $P\text{-}PO_4^{3-}$), and bacteria E. coli, and E. intestinal, all linked with organic pollution. This addresses the fact that the water samples need a chemical treatment by $H_2O_2$ to destroy organic matter. Water samples revealed low salinity. For better separation of MPs and TSS, the salinity of these samples was adjusted to 25% NaCl.

*Identification by microscope analysis*

Microscopic analysis enabled the classification of MPs based on characteristic colour, shape, texture, and morphological features (Figure 2). The reports for each sample are given in Table 2.

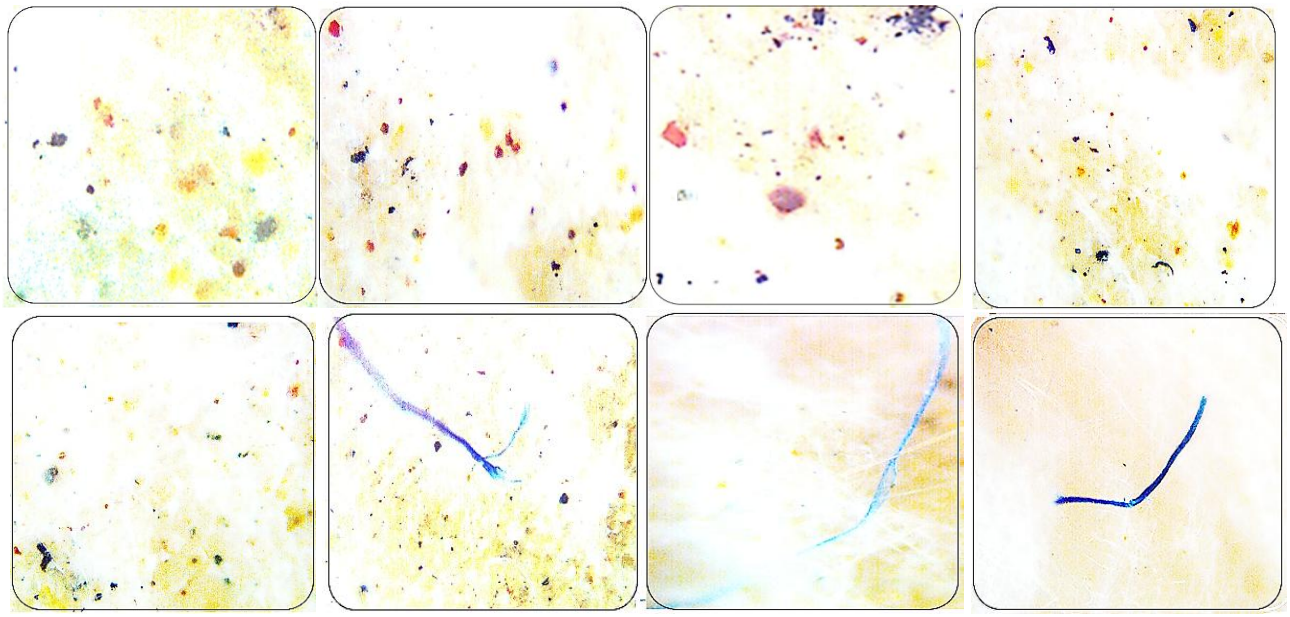

**Fig. 2** Microscopic images performed in five points of Watman filter after sample filtration

**Table 2**. Characteristic features of MPs' optical microscopic images from collected samples

| Colour | Physical character | Classification |
|---|---|---|
| Blue, dark grey, red, yellow, green | Fibers, small particles | PCP, PE, rubber 3, PVC, PS |

PCP – Polychloroprene; PE – Polyethylene; PVC - Polyvinyl chloride; PS – Polystyrene

The morphological characteristics and textures examined via microscopic images exhibit substantial variation, marked by heterogeneity in colour, shape, texture, and thickness. The classification of MPs was carried out utilizing the polymer cluster map developed by Helmholtz [33]. The findings presented are preliminary in nature and require additional confirmation, which was achieved through FTIR analysis. However, they are consistent with previous studies conducted in the Durres Bay [34] under the framework of the GEF-funded project (2018÷2021). According to Milica Mandić [34], the average number of marine litter items for five Dures Bay beaches including Shen Pjetri, Apolonia,

Iliria, Dajlani, and Golem beaches, was 0.14 items/m$^2$ (333 items/100 m stretch). Based on the clean coastal index (CCI) all five beaches belong to „clean "beaches (CCI = 1.8÷4.2). The majority of litter items (more than 90%) were made out of artificial polymer materials, followed by rubber, cloth/textile, paper/cardboard, and glass/ceramics.

*FTIR analysis of microplastics in water samples of Erzeni River estuary*

Characterization and identification of MPs present in the sample were performed by FTIR analysis within the region of 4000 cm$^{-1}$ to 400 cm$^{-1}$. The FTIR spectra of water samples of this study are shown in Figure 3.

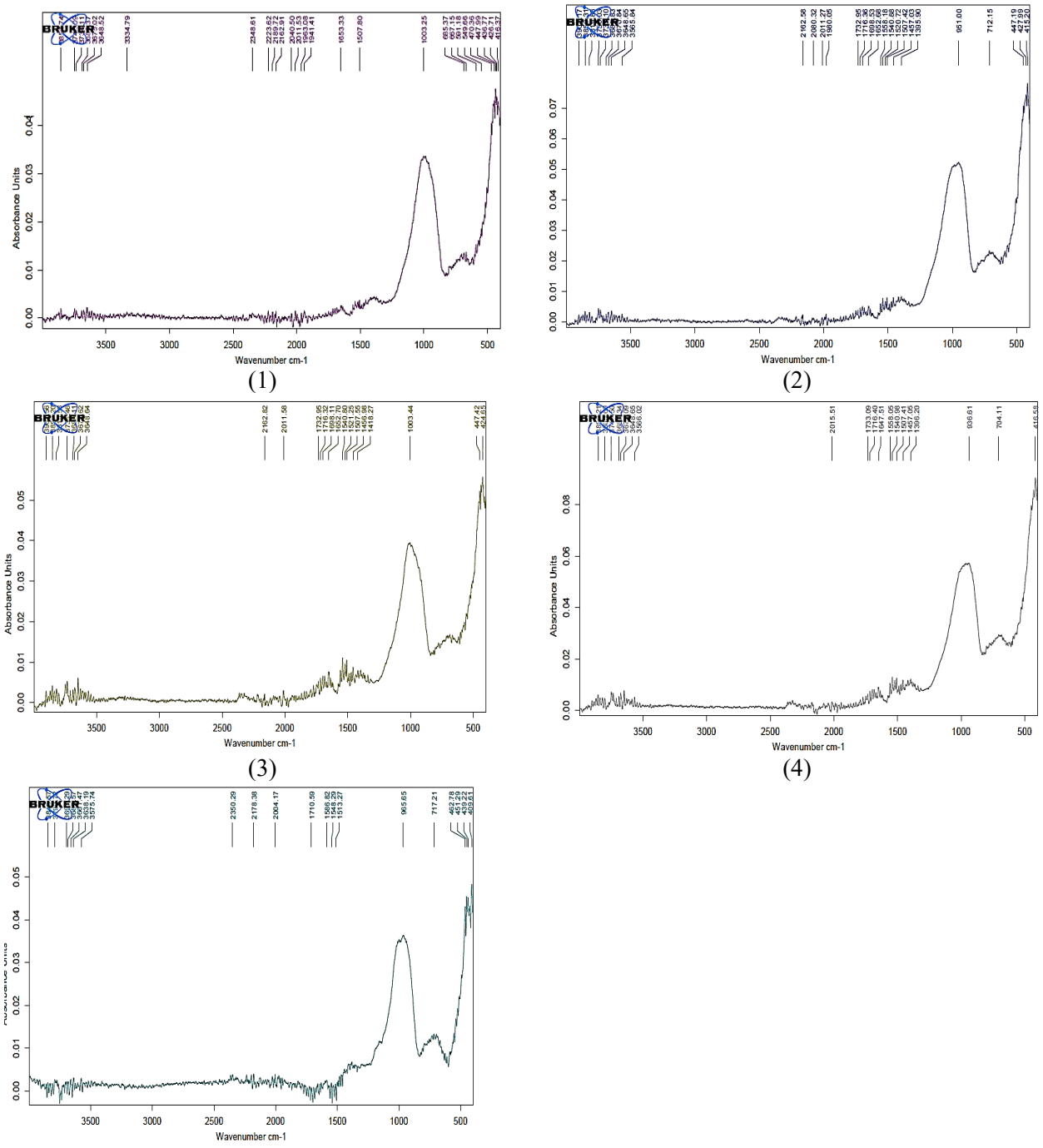


**Fig. 3.** FTIR spectra of the filter collected at five points near the mouth of the Erzeni River

The FTIR spectra obtained from the analysis of the water sample at the mouth of the Erzeni River exhibit consistent characteristics, with major spectral bands appearing in similar regions across

samples. These similarities underscore the presence of key molecular features. The most prominent bands, distinguished by their high intensity, were observed within the 600÷1000 $cm^{-1}$ range. This spectral region, commonly known as the fingerprint region, is indicative of specific polymers and is associated with the vibrational modes of C–C and C–H bonds, among other functional groups [35].

Table 3 summarizes the principal spectral bands identified from measurements taken at five distinct locations on the filter collected at five points near the mouth of the Erzeni River.

**Table 3.** FTIR bands (in $cm^{-1}$) measured at filters from the discharge area of the Erzeni River

| Region | 3900÷3500 | 2200÷2000 | 2000÷1600 | 1600÷1400 | 1400÷1300 | 1000÷700 | 500÷400 |
|---|---|---|---|---|---|---|---|
| Point 1 | 3648, 3675, 3689, 3734, 3746, 3854 | 2349, 2224, 2190, 2163, 2041, 2012 | 1963, 1941, 1653 | | | 1003, 690, 668 | 591, 549, 470, 448, 437, 427 |
| Point 2 | 3902, 3853, 3820, 3750, 3735, 3690, 3671, 3649, 3566 | 2163, 2080, 2011 | 1980, 1733, 1716, 1699, 1653 | 1558, 1541, 1521, 1507, 1457, 1396 | 1457, 1396 | 951, 712 | 447, 428, 415 |
| Point 3 | 3903, 3853, 3820, 3734, 3689, 3675, 3649 | 2163, 2012 | 1733, 1716, 1698, 1653 | 1541, 1521, 1508 1457, 1418 | | 1003 | |
| Point 4 | 3853. 3801, 3750, 3689, 3675, 3649, 3566 | 2016 | 1733, 1716, 1648 | 1558, 1541, 1507, 1457, 1396 | | 937, 704 | 417 |
| Point 5 | 3847, 3791, 3697, 3684, 3661, 3638, 3576 | 2350, 2178, 2004 | 1711 | 1587, 1548, 1513 | | 966, 717 | 463, 451, 439, 410 |

The identified key bands of different regions can be interpreted as follows:
- The bands observed at approximately 3648 $cm^{-1}$, 3675 $cm^{-1}$, 3689 $cm^{-1}$, 3734 $cm^{-1}$, 3746 $cm^{-1}$, and 3854 $cm^{-1}$ could be interpreted as broad bands of O–H/N–H stretching of cellulose and/or nylon particles [36].
- The next bands positioned at approximately ~3335 $cm^{-1}$ could be interpreted as broad bands of O–H stretching vibrations of carboxylic acid polymers, like PS [36].
- The band at 2349 $cm^{-1}$ likely derived from atmospheric $CO_2$, which shows the theoretical peak at around 2350 $cm^{-1}$, and the bands observed at approximately 2349 $cm^{-1}$, 2224 $cm^{-1}$, 2190 $cm^{-1}$, 2163 $cm^{-1}$, 2041 $cm^{-1}$, and 2012 $cm^{-1}$ lie within the region of ~2324÷1981 $cm^{-1}$ that can indicate isocyanates or carbonyl groups in modified plastics [36].
- The weak bands stretched between 2000 $cm^{-1}$ and 1650 $cm^{-1}$, such as 1963 $cm^{-1}$, 1941 $cm^{-1}$, and 1653 $cm^{-1}$, could be interpreted as C-H bending vibrations of aromatic compounds [36].
- FTIR spectra within the range of 1400 $cm^{-1}$ to 1600 $cm^{-1}$ display typical bands associated with C-H bending vibrations and C=C stretching vibrations found in aromatic rings. They offer valuable insights into the molecular structure of the MPs identified in the analysed sample. The bands present in this region are crucial for characterizing the types of MPs present in the sample, especially for polymers like PS and polyethylene terephthalate (PET).
- The peaks at 1000 $cm^{-1}$ to 416 $cm^{-1}$ could be interpreted as halogenated plastics, like PVC, or other inorganic fillers.
- FTIR absorption bands in the region of 1000 $cm^{-1}$ to 600 $cm^{-1}$ can be used to identify specific polymers containing C-O, C-C, or C-Cl bonds, identifying the PE, PS, and/or PVC. The strong peaks at 1003 $cm^{-1}$, 690 $cm^{-1}$, and 668 $cm^{-1}$ may show C-O, C-C, or C-Cl stretching or the presence of the

O-Si-O band of montmorillonite-modified (MMT) plastics. Absorption bands in this region can also be used to identify the C=C bonds or aromatic rings [37].
- The region between 400 $cm^{-1}$ and 600 $cm^{-1}$ is often associated with skeletal vibrations. The peaks at 591 $cm^{-1}$, 549 $cm^{-1}$, 470 $cm^{-1}$, 448 $cm^{-1}$, 437 $cm^{-1}$, 427 $cm^{-1}$, and 416 $cm^{-1}$ may be related to the multiple peaks of C-Cl bonds of PVC or other specific polymer structures and may be interpreted as a fingerprint of a specific plastic polymer or could indicate the presence of the halogenated polymers, such as PVC, as well as the MMT plastics. It is supported by the strong absorbance in this region.
The bands at the region of 400 $cm^{-1}$ to 1100 $cm^{-1}$ could be linked with MMT, known as synthetic rubber [37]. Beside the strong key bands at the region of 400 to 1100 $cm^{-1}$, the presence of MMT is verified also by microscopic images (features in deep gray). This synthetic rubber is characterized by high durability and environmental and chemical resistance, making it a preferred material for both industrial applications and consumer goods [36÷38].
According to the analysis above, the water samples may contain PVC or PVC/MMT, nylon, PS, and PE contaminants. PVC in environment originates various sources, such as manufacturing, the breakdown of other chemicals, improper disposal, and/or leaks. PVC is a harmful plastic for the environment and human beings [39]. The full cycle of PVC production, utilization, and disposal leads to the emission of the toxic chlorine-derived chemicals, making it one of the leading sources of dioxins on a global scale, which accumulate in water, air, and food chains [39].
PE is a widely used plastic in the packaging industry [40, 41], greenhouses, wire insulation, children's toys, and other industrial and everyday applications. The past two decades have seen a significant increase in plastic usage, resulting in a corresponding increase in plastic waste, which is often disposed of in landfills, incinerated, recycled, or released into the environment [40, 41]. The persistence and non-biodegradable nature of PE made it a pressing challenge, posing a serious threat to the environment, biodiversity, and human health.
Albania is working to improve legislation on waste management, including microplastic waste, with the aim of aligning it with EU standards and implementing a strategy to reduce plastic pollution. While the country faces challenges such as limited separate waste collection systems, the focus is on developing best practices for waste management. In this context, Albania has adapted the legal structures set by the EU directives on waste management and has established a National Plan for Integrated Waste Management (2020÷2035) that includes specific goals for recovering plastic packaging waste. In addition, the target of the National Waste Management Strategy 2021 of Albania aims to reduce plastic waste by 70% and improve waste management infrastructure.

## CONCLUSIONS

This study was carried out in the delta of the Erzeni River, along the Adriatic Sea in Albania. It represents a preliminary qualitative investigation into the presence and types of microplastics (MPs), focusing on the impact of river water on microplastic pollution in the adjacent coastal area. Identification of the types and presence of MPs in water samples was performed through microscopic examination and FTIR analysis. Microscopic images revealed diverse morphological features, including variations in color, shape, texture, and thickness, indicating the presence of multiple microplastic types.
FTIR analysis detected PVC and/or modified PVC, nylon, PS, and PE as the predominant microplastic polymers within this ecosystem. Microscopy showed a limited number of MPs in one-liter water samples, which correlated with FTIR spectra characterized by low absorbance and high noise levels. Furthermore, the coexistence of various MP types in the samples likely contributed to increased spectral interferences, complicating accurate identification.
To obtain stronger spectral signals and more detailed microscopic imagery, we recommend increasing the volume of water samples collected for analysis from 1 liter to 5 liters. Another challenge concerns maintaining the integrity of the sample matrix during the density separation step. The commonly used NaCl solution achieves a maximum density of approximately 1.2 $g/cm^3$, which is insufficient for separating microplastics with higher densities, such as PVC, which has a maximum density of 1.45 $g/cm^3$. Therefore, we suggest exploring the use of higher-density solutions, such as sodium bromide

(NaBr) or ferric chloride ($FeCl_3$), which can significantly increase the density of aqueous solutions and improve the efficiency of density separation.

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