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Monitoring of Microplastics in Water and Sediment Samples of Lakes and Rivers of the Akmola Region (Kazakhstan)

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Abstract: This paper provides a detailed description of the findings and methodology related to the monitoring of microplastics in three lakes and one river of the Akmola Region in Kazakhstan. The concentration of microplastic particles and the analysis of water and sediment quality of the Yesil River and Kopa, Zerendinskoye, and Borovoe lakes have been analyzed. A total of 64 water samples were collected across the spring, summer, and autumn seasons, with subsequent analysis revealing a seasonal increase in microplastic concentrations. The average microplastic content ranged from 1.2×10^{-1} particles/dm^3 in spring to 4.5×10^{-1} particles/dm^3 in autumn. Lakes exhibited higher concentrations compared to the Yesil River. Correlation analysis highlighted a connection between microplastic content and turbidity, particularly notable during the spring season. Analysis of sediments revealed a decrease in microplastic concentrations from the coastal zone toward open waters sediments. Microplastic fibers were predominant in sediments (69.6%), followed by fragments (19.1%), films (7.4%), and granules (3.9%). Larger particles (>500 µm) were found in beach sediments, constituting an average of 40.5% of the total plastics found. This study contributes valuable insights into the spatial and temporal distribution of microplastics, emphasizing the need for ongoing monitoring and management strategies to address this environmental concern.

Keywords: microplastics; Akmola region; Yesil river; sediments; water quality indicators

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1. Introduction

Improved water supply, sanitation, and better water resource management can boost economic growth and aid in poverty reduction. Access to clean and safe water is crucial for maintaining a healthy society [1]. However, the increasing presence of suspended organic and inorganic substances, along with anthropogenic macro and micro ions in water bodies, hampers natural self-purification processes and harms aquatic life. Elevated levels of these substances in watercourses can lead to severe diseases and health issues in humans [2–4].

This study builds upon extensive scientific research conducted in Northern Kazakhstan, specifically in the North Kazakhstan and Akmola provinces. Previous investigations focused on protecting and efficiently utilizing water resources, as well as evaluating water quality [5–7]. Both natural and human-induced factors affecting hydrochemical indicators in Northern Kazakhstan's natural waters were explored. Long-term studies have shown that anthropogenic activities significantly impact hydrochemical indicators in surface waters, contributing to their deterioration [8–11].

The challenges surrounding the management and processing of solid domestic waste persist as critical and unresolved issues in contemporary Kazakhstan. Recent studies have examined various aspects, including the sorting and utilization of solid domestic waste [12–14], the recycling of plastic waste [15,16], and the impact of plastic bottle landfills and dumps on environmental components [17–19].

Plastics are widely manufactured and utilized for various purposes, ranging from medical and technological applications to packaging and wrapping, owing to their unique properties such as low density, thermal and electrical conductivity, corrosion resistance, and cost-effectiveness. However, despite its widespread use, plastic has evolved into a global menace for the natural environment [20], human health, and living organisms. The convenience of plastic has led to approximately 40% of it being single-use with a brief lifespan of just a few minutes but a decomposition period in the environment that can extend to several hundred years [21]. According to [22], "2500 million tons of plastic, equivalent to 30% of all plastics ever produced, are currently in use. Between 1950 and 2015, the cumulative generation of primary and secondary (recycled) plastic waste was 6300 million tons. Of this, about 800 million tons (12%) of plastic were incinerated, and 600 million tons (9%) were recycled, with only 10% of it being recycled more than once".

Due to their low density, which is similar to that of water, plastic microparticles tend to float on the water's surface. The y are easily transported by surface runoff from coastal areas, landfills, and unauthorized dumps into rivers and lakes. This situation is particularly severe in regions with high tourism potential and a poor culture of plastic waste disposal, leading to the littering of coastal zones with plastic debris. Inadequate infrastructure and a lack of environmental awareness among the population and tourists in the Akmola region and Kazakhstan as a whole contribute to the widespread pollution of coastal areas, which then extends to rivers and lakes. The problem is exacerbated by numerous unauthorized landfills and the discharge of household wastewater into lakes, increasing the risk of plastic pollution in water bodies. According to reports from the Ministry of Ecology, Geology, and Natural Resources of the Republic of Kazakhstan and Forbes Kazakhstan, satellite images taken in 2021 identified over 600 unauthorized solid waste disposal sites in the Akmola region, with the regional center of Kokshetau accounting for 23.5% (147 dumps). The region lacks proper facilities for sorting and processing solid domestic waste, resulting in a recycling rate of no more than 3% [23].

The absence of effective plastic recycling solutions results in the gradual degradation of plastic waste in the environment due to solar radiation, mechanical forces, and biological processes, forming particles of various sizes, including macro-, micro-, and nano-sized, known as microplastics (MP) [19]. While there are ongoing discussions regarding the definition and classification of microplastics, as well as the lower size limit, it is widely accepted that they encompass plastic particles smaller than 5 mm [24–27]. The se small plastic particles pose significant environmental hazards and can enter the bodies of animals and humans through water and food intake [28–30]. Studies suggest that Americans consume an estimated 13% of their daily diet in the form of microplastic particles. Additionally, previous research has examined drinking water as a potential source of microplastic ingestion. A study conducted by Newcastle University for WWF in 2019 found that, on average, individuals may ingest up to 5 g of plastic per week through food consumption [31].

In 2017, it was reported that microplastic (MP) particles can accumulate in the liver, kidney, and intestine of mammals, with the rate of tissue accumulation and distribution influenced by the particle size. Exposure to microplastics has been observed to disrupt energy and lipid metabolism, as well as induce oxidative stress [32]. In 2019, a study highlighted the inadvertent ingestion of microplastics by humans from various sources. The authors underscored the need for further research into the extent of microplastic consumption and its potential impact on human health [33]. Additionally, the presence of microplastics has been detected in all parts of the placenta, including maternal, fetal, and amniochorionic membranes. This suggests that microplastics carry substances that act as endocrine disruptors, potentially leading to long-term health effects [34].

The danger of microplastic (MP) intake into the bodies of animals and humans is also linked to its ability to absorb and hinder the sedimentation of organic pollutants and heavy metals, leading to increased turbidity and coloration of natural water.

Pollutants absorbed in this manner can subsequently enter the bodies of aquatic organisms and humans, elevating the risk of toxic effects [35–37]. The significance of international research on microplastics as an environmental pollutant is evident from the substantial increase in scholarly works over the past decade. Due to the relatively limited study of this issue, research topics are diverse, encompassing analyses of extent, behavior, risks to human health and specific species, and the impacts of microplastic pollution in freshwater systems [38]. Other areas of research include the standardization of monitoring methods in marine regions to facilitate comparison and assessment of microplastic pollution over time [39], as well as the historical development of marine anthropogenic litter [40]. Analysis of scientific literature available in open sources indicates a lack of research on microplastics in Kazakhstan's environment, with existing publications often lacking in-depth analyses of the problem. For instance, scientists at the Institute of Hydrobiology and Ecology of the Republic of Kazakhstan conducted an analysis of macro- and microplastics based on monitoring results from the Caspian Sea. The ir study concludes that further research is necessary to qualitatively and quantitatively assess the extent of plastic pollution and its impact on living organisms and the ecosystem of the sea [41].

One of the few studies conducted in Russia, a neighboring country to Kazakhstan, was published in 2021, reporting for the first time on microplastics in inland lakes of South Siberia. This study investigated particle sizes, their concentrations in lakes, surface morphology, and the elemental composition of plastics using spectroscopic methods. Additionally, factors influencing the presence of microplastics in lakes were identified. The paper emphasizes the necessity of increasing attention to waste management practices [42]. The quantity and distribution of microplastics in natural waters can be influenced by various environmental factors, including salinity, water depth, pH, and temperature [43]. However, further research with additional factors and experimental data are required to establish their impact on the prevalence of microplastics in natural waters [44]. Given the lack of comprehensive studies on microplastics in natural waters of Kazakhstan, it is pertinent to monitor microplastics in rivers and lakes, seek correlations between microplastic content, water depth, and quality, and analyze microplastic content in contact media such as coastal and bottom sediments.

In this work, the behavior of microplastics in the natural water objects of the Akmola region (Kazakhstan) is analyzed, giving recommendations on the sustainable and safe use of natural waters in Kazakhstan. This research helps to find a solution to the issue of the safety of natural waters in the region while contributing to the study of the problem of pollution of natural waters by microplastics.

2. Description of the Study Area

Microplastic monitoring was conducted within the Yesil River basin, the sole watercourse in the Akmola region of Kazakhstan, running from southeast to northwest and further north towards the North Kazakhstan region [45]. The Akmola region encompasses a 1027 km-long section of the river, covering approximately 20,000 km^2 [46]. The formation of a stable ice cover typically occurs by the end of November, with ice formation potentially beginning as early as October. The river experiences significant spring flooding, which typically subsides by the end of May in the upper reaches and by the end of June in the northern reaches [47]. Based on these characteristics, sampling was scheduled during the spring period (at the end of the spring flood) in May and during the autumn period (before ice cover formation) in September. Summer precipitation has minimal impact on the river's water regime, and summer floods are uncommon [48]. During the sampling period in the summer season of 2023, no rainfall was observed.

The Yesil River within the city of Astana, the capital of Kazakhstan, is a popular destination for both local residents and visitors. The water quality of the Yesil River in Astana is influenced by various factors in the sampling area, including industrial enterprises such as the "GazMashApparat" plant, overflow dams, river tributaries (such as the Akbulak river mouth), Triathlon Park, and city embankments frequented by the population.

Additionally, within the Akmola region, lakes Kopa, Zerendinskoye, and Borovoe were examined for microplastic content. The se lakes are all heavily visited by the population, with Zerendinskoye and Borovoe Lakes being particularly popular during the summer months. Lake Kopa, located within the city of Kokshetau, sees visitors throughout the year. The locations of these three lakes are indicated in Figure 1.

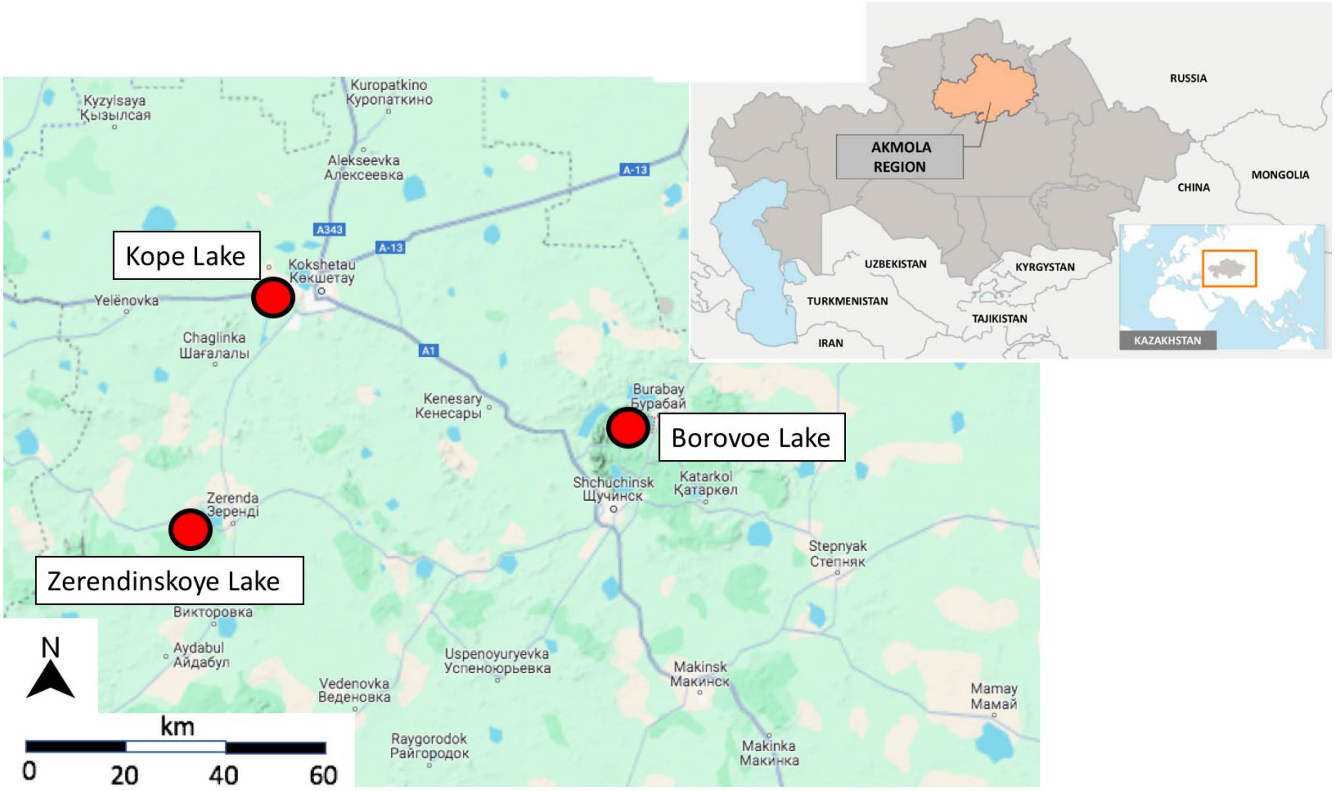

Figure 1. Location of Kopa, Zerendinskoye and Borovoe lakes.

Lake Kopa is situated near the base of the Kokshetau Upland, in the northwestern part of Kokshetau City. It spans an average area of 14 km^2 with depths ranging from 2.0 to 3.0 m. The total catchment area is 3860 km^2, primarily consisting of the lake's tributaries: the Chaglinka River from the southwest and the Kylshakty River from the southeast, with only a small portion (80 km^2) directly contributing to the lake [49]. The lake remains perennial and does not dry up. The water quality of Lake Kopa may be influenced by various factors including public groundwater wells, the Urker Palace restaurant, the Shagalaly River which flows into the lake carrying pollution from Krasny Yar village, as well as the presence of railway tracks and the city beach. Sampling sediment was challenging due to dense reed and cattail thickets, with an average width of 300 m, and a viscous lake bottom covered with a layer of clayey, loamy silt ranging from 0.5 to 2.8 m thick.

Lake Borovoe, located in the Shchuchinsky district of the Akmola region, is a drainless lake situated at the eastern base of Mount Kokshe. Covering an area of 10.5 km^2 (with dimensions of 4.5 km in length and 3.9 km in width), it boasts an average depth ranging from 4.5 to 7.0 m [50,51]. Lake Borovoe serves as a resort destination, frequented not only by local residents but also by tourists from distant regions of Kazakhstan and abroad. The water quality of Lake Borovoe may be affected by various tourist attractions, including the boat station, Baitas Hotel, S. Seifullin Secondary School, the ring transport road, the mouth of the Sarybulak Brook, and the Aynakol entertainment complex.

Lake Zerendinskoye, located in northern Kazakhstan within the Yesil River basin, is approximately 50 km away from Kokshetau. It measures 5.3 $\times$ 3.5 km in size, with an area of 9.61 km^2 and a coastline spanning 19.4 km. The lake boasts an average depth of

4.2 m and features a flat sandy bottom, occasionally adorned with pebbles and scattered boulders. Sandy beaches adorn the shores, and the water of the lake is fresh and transparent. While Lake Zerendinskoye is not a popular tourist destination, certain areas along its shoreline may pose a risk to water quality. The se include the M. Gabdulin secondary educational school, a rural hospital, the Zeren Nur recreation center, a fishery enterprise, the Vostochnaya recreation center, and a public recreation area near the Balkadisha monument.

3. Materials and Methods

3.1. Location of the Sampling Points

The selection of natural water sampling points was carried out in collaboration with specialists from the Astana and Kokshetau branches of the Republican State Enterprise "KazHydromet". During the selection process, consideration was given to potential sources of pollution and the key characteristics of the rivers and lakes under investigation. Detailed information regarding the sampling points, as well as the main characteristics of the water bodies, can be found in Tables 1–4 and Figures 2–5.

Table 1. Sampling points at Kopa Lake.

Sampling Point	GPS Coordinates	Description
1	53.286948, 69.354102	Public groundwater wells
2	53.297121, 69.336914	Urker Palace Restaurant
3	53.313779, 69.322168	Mouth of the Shagalaly River (Krasny Yar village)
4	53.327312, 69.328756	Railway tracks (Krasnoye village)
5	53.319875, 69.364748	Kokshetau meteorological station "Kazhidromet" for Akmola region
6	53.300309, 69.379781	City beach
7	53.312930, 69.351676	Center of the lake
8	53.305157, 69.357763	Center of the lake

Table 2. Sampling points at Lake Zerendinskoye.

Sampling Point	GPS Coordinates	Description
1	52.914085, 69.141020	M. Gabdulin Secondary School
2	52.906375, 69.126523	Rural hospital
3	52.922582, 69.083956	Zeren Noor Recreation Centre
4	52.940660, 69.113021	Fishery enterprise
5	52.940180, 69.125558	Vostochnaya recreation center
6	52.935238, 69.136085	Public recreation site, Balkadisha Monument
7	52.928451, 69.121305	Center of the lake
8	52.920951, 69.127372	Center of the lake

Table 3. Sampling points at Lake Borovoe.

Sampling Point	GPS Coordinates	Description
1	53.089900, 70.254179	Boat station
2	53.087245, 70.268173	Hotel Baitas
3	53.084186, 70.299261	S. Seifullin Secondary School
4	53.076950, 70.302128	Ring Road
5	53.067756, 70.301272	Mouth of Sarybulak Creek
6	53.061712, 70.296819	Aynakol Entertainment Complex
7	53.078176, 70.278866	Center of the lake
8	53.071580, 70.280757	Center of the lake

Table 4. Sampling points on the Yesil River (within Astana city limits).

Sampling Point	GPS Coordinates	Description
1	51.159911, 71.398368	GasMashApparat plant area
2	51.156111, 71.407128	Overflow dam
3	51.160420, 71.422823	City embankment, area of Kenesary monument location
4	51.149496, 71.437259	Mouth of Akbulak River
5	51.132982, 71.447133	Triotlon Park
6	51.124678, 71.453330	City embankment, the area of the residence of the President of the Republic of Kazakhstan Akorda
7	51.152855, 71.427425	Riverbank
8	51.159903, 71.417597	Riverbank

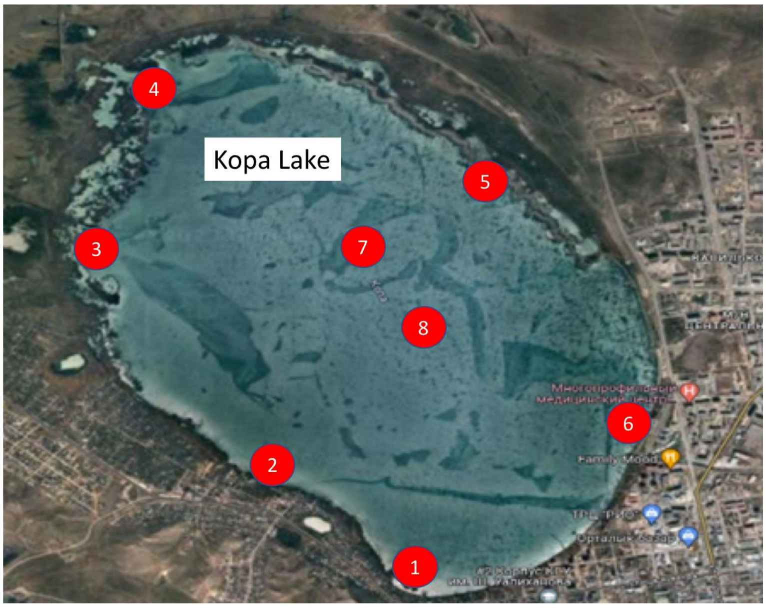

Figure 2. Map-scheme of sampling points location at Kopa Lake.

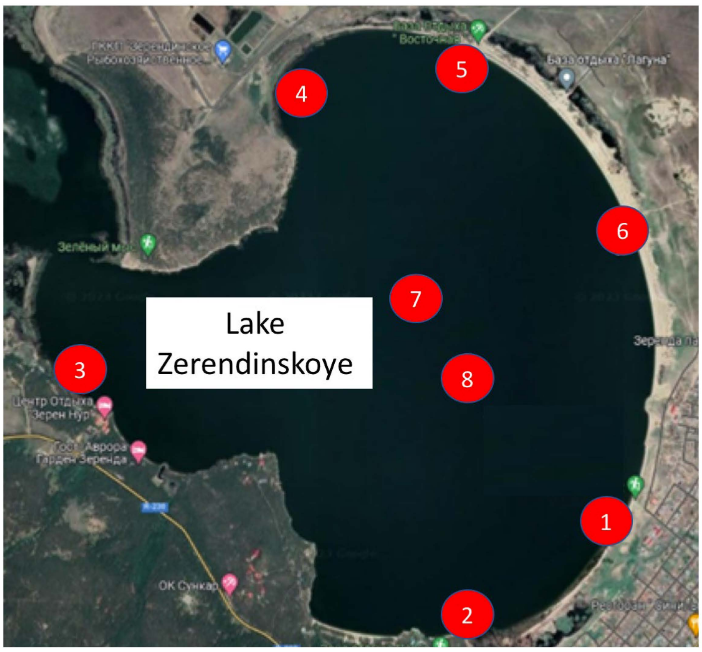

Figure 3. Map-scheme of sampling points location at Lake Zerendinskoye.

Figure 4. Map-scheme of sampling points location at Lake Borovoe.

Figure 5. Map-scheme of sampling points on the Yesil River within the city of Astana.

For sediment sampling, a total of 30 sampling points were identified, and their coordinates were meticulously recorded. The sampling points were categorized as DO (denoting bottom sediments), SW (representing the water's edge), and ZZ (indicating the splash zone). Detailed information regarding these sampling points, along with their respective coordinates, can be found in Tables 5–8.

Table 5. Sediment sampling points at Kopa Lake.

Sample Number	GPS Coordinates	Location
1 2 3	53.300309, 69.379781	DO UW ZZ
4 5 6	53.297121, 69.336914	DO UW ZZ

Table 6. Sediment sampling points at Lake Zerendinskoye.

Sample Number	GPS Coordinates	Location
7 8 9	52.940180, 69.125558	DO UW ZZ
10 11 12	52.935238, 69.136085	DO UW ZZ
13 14 15	52.914085, 69.141020	DO UW ZZ

Table 7. Sediment sampling points at Lake Borovoe.

Sample Number	GPS Coordinates	Location
16 17 18	53.076950, 70.302128	DO UW ZZ
19 20 21	53.067756, 70.301272	DO UW ZZ
22 23 24	53.061712, 70.296819	DO UW ZZ

Table 8. Sediment sampling points on the Yesil River.

Sample Number	GPS Coordinates	Location
25 26 27	51.156111, 71.407128	DO UW ZZ
28 29 30	51.101215, 71.514357	DO UW ZZ

3.2. Water Sampling Equipment and Sampling Methodology

The following equipment was utilized for water sampling to analyze physico-chemical parameters and for the extraction of microplastics and sediment sampling:

- GR-91 rod dredger with a bucket volume of 300 cm^3;
- Ruttner bathometer with a volume of 5 dm^3;
- Metal bucket with a volume of 10 dm^3;
- Metal scoops and spatulas;

- Glass jars;
- Glass Petri dishes;
- Metal tweezers;
- Aluminum foil;
- Sefar polyamide mesh with a mesh size of 100 μm.

For sampling surface water sources for microplastic analysis, a metal bucket with a volume of 10 dm^3 was used. Water was collected by scooping from the surface layer at a depth ranging from 5 to 20 cm. Filtering was conducted until a total volume of 100 L (equivalent to 10 buckets of 10 dm^3) of water was filtered.

Water was filtered from a depth of 1.5 m using a bathometer with a volume of 5 dm^3, with a total volume filtered from this depth amounting to 50 dm^3. The study refrained from using manta trawl nets due to concerns regarding the potential for cross-contamination and microplastic transfer between samples [52]. To mitigate this risk, a new filter was employed for each sample. Despite the widespread global practice of using meshes ranging from 300 to 390 μm for monitoring microplastics in surface waters [53–55], the study opted for the smallest mesh size available in Kazakhstan, which was 100 μm. Volumetric sampling, followed by filtration similar to the approach outlined in [56], was employed in the study. At each new sampling point, the sampler, bucket, and filter device were thoroughly rinsed with distilled water. Following filtration, the filters were transferred to glass Petri dishes, with each filter placed in a separate dish labeled with the sample number and location.

Recent estimates indicate that 70% to 90% of aquatic microplastic particles accumulate in sediment profiles [57]. To validate the hypothesis of microplastic input into water bodies from the coastal zone and to gain a more comprehensive understanding of microplastic distribution, the content of microplastics in bottom sediments of open waters and coastal zone sediments was assessed at three distinct locations [58]:

(i) Wave splash zone;
(ii) Water edge;
(iii) Bottom sediment zone at depths ranging from approximately 1.23 to 1.53 m (the maximum feasible depth for manual sediment sampling, determined by the geological structure of the bottom).

In each zone, samples were collected at five evenly distributed points along the entire 10-m length of the site. This method resulted in a total of five individual samples for each zone, covering bottom sediments, the water's edge, and the splash zone. The boundaries of the sampling zones were delineated using a measuring tape. Cut-off and sediment samples were gathered with a metal scoop into glass jars, while bottom sediments were obtained using a hand dredge. Accessible sites were chosen for sediment sampling to ensure the collection of all three intended sediment groups: sediments, the water's edge, and the wave splash zone. Sampling was avoided in areas where shoreline access was impractical, such as locations with overgrown reeds or areas where the natural shoreline had been altered into a stone embankment.

Bottom sediments were sampled to a depth of 5.0 ± 0.5 cm and packed in glass containers with metal lids. Each sample was carefully labeled and transported to the laboratory for further analysis. Upon arrival, the samples were dried at 30 °C and subsequently stored in glass containers in a refrigerator at 4 °C until analysis. Beach sediments (referenced in Table 9) were sampled from an area located on the same transect (perpendicular) as the sampled sediments. This area was positioned at a far distance of 5 m from the water's edge line, extending 5 m from the perpendicular line in each direction. The defined area was marked with colored tape, with a total area of 10 × 5 m.

Table 9. Beach sediment sampling points.

Sample No.	Place of Selection	GPS Coordinates
1	Kopa Lake	53.300309, 69.379781
2	Kopa Lake	53.297121, 69.336914
3	Zerendinskoye Lake	52.940180, 69.125558
4	Zerendinskoye Lake	52.929259,69.142834
5	Zerendniskoye Lake	52.914085, 69.141020
6	Borovoe Lake	53.076950, 70.302128
7	Borovoe Lake	53.067756, 70.301272
8	Borovoe Lake	53.061712, 70.296819
9	Yesil River	51.101215, 71.514357

3.3. Water Quality Standards

All water samples collected during the spring, summer, and autumn seasons were subjected to analysis for 10 physical and chemical indicators as per the regulations outlined in the Order of the Minister of Health of the Republic of Kazakhstan dated 24 November 2022, No. KR DSM-138, titled "On approval of hygienic standards of safety indicators for household and cultural and domestic water use" [59]. The methods utilized for analyzing the physico-chemical parameters of water reservoirs are detailed in Table 10.

Table 10. List of the main measured indicators of water quality and methods for their determination.

Indicator	Method of Measurement	Detection Limit	Standards and References
Color degree	Photometric	±1 degree	Interstate standard 31868-2012. Water. Methods for determining color [60]
Turbidity	Photometric	±0.01 mg/dm^3	Interstate standard 3351-74. Drinking water. Method for determination of odor, taste, color, and turbidity [61]
pH	Potentiometric	±0.01 unit	RK ISO standard 4316-2019. Surfactants. Determination of pH of aqueous solutions. Potentiometric method [62]
Oxidizability (COD)	Titrimetric	±0.1 mg/dm^3	State mandatory standard 26449.1-85. Stationary distillation and desalination plants. Methods for chemical analysis of salt waters [63]
Ammonium ions	Photometric	±0.01 mg/dm^3	Interstate standard 33045-2014. Water. Methods for determining nitrogen-containing substances [64]
Hardness (concentration of calcium and magnesium ions)	Titrimetric	±0.01 mg/dm^3	Interstate standard 31954-20120. Drinking water. Methods of hardness determination [65]
Mineralization (dry residue)	Gravimetric	±1 mg/dm^3	State mandatory standard 18164-72. Drinking water. Method for determination of total solids content [66]
Sulfates	Titrimetric	±0.1 mg/dm^3	Interstate standard 4389-72. Methods for determination of sulfate content [67]
Total iron	Photometric	±0.01 mg/dm^3	Interstate standard 4011-72. Drinking water. Methods for determination of total iron [68]
Carbonates	Titrimetric	±1 mg/dm^3	State mandatory standard 26449.1-85. Stationary distillation and desalination plants. Methods for chemical analysis of salt waters [63]

3.4. Methodology to Extract and Analyze Microplastics from Water and Sediment Samples

To extract and analyze microplastics from water and sediment samples, the following equipment was used:

- Microscope: DTX 500 LCD Levenhuk with photo and video registration;
- Analytical electronic scales: AX-200 Shimadzu (measurement accuracy 0.0001 g);
- Stainless steel sieves with mesh sizes: 3, 2, 1, 0.3, 0.175 mm;
- Electric dry-air thermostat: TS-1/80 SPU (maximum deviation of the average temperature not more than ±1 °C, maximum deviation of the temperature at any point ±0.4 °C);
- Water bath: "Ekros" model 4310;
- Ultrasonic bath: UZV-4.0 "Sapphire" with a digital thermostat (temperature range from 15 to 70 °C, ultrasound frequency 35 kHz, timer from 1 to 99 min);
- Filters for quantitative analysis: Whatman No.2;
- Laboratory centrifuge: Opn-3.01 "Dastan" centrifuge with rotation speeds of 1000, 1500, and 3000 rpm;
- Set of areometers;
- 5.75 M ZnCl solution.

Based on the analysis of 100 sources [69], and their adaptation to laboratory conditions, we developed a working protocol for extracting and separating microplastic particles from organic substances and foreign impurities in both aqueous and solid media. The quality of the research was ensured by implementing the "negative" control method to prevent cross-contamination of samples with plastic. The efficiency of microplastic extraction was evaluated using the "positive" control method [70,71]. Measures were taken, as suggested by [72,73], to prevent cross-contamination of samples with microplastics. To prevent contamination from the air, most of the work was conducted in a fume cupboard, and the analyzed filters were consistently stored in glassware, specifically Petri dishes with a closed lid [74]. Throughout all experiments, synthetic materials were avoided, and only cotton lab coats and metal instruments were used [75]. Before each session, all instruments were washed with distilled water, and surfaces were cleaned with ethyl alcohol, followed by wiping with distilled water.

A "negative" control was implemented by checking materials (Petri dishes, polyamide filters, flasks, beakers, bottles, and tables) for the presence of microplastics. For this control, polyamide filters pre-wrapped in foil and aged in muffle ovens at a temperature of 350 °C were utilized. Additionally, weekly checks of clean filters in labeled Petri dishes with an open lid were conducted.

Positive controls were implemented to prevent the loss of microplastic particles at different stages of extraction from the sample [76–78], involving the addition of UV fluorescent microplastic particles of various fractions. In the laboratory, filters used for the filtration of natural waters without visually noticeable organic contamination were dried either in a desiccator at room temperature or in a desiccator at 35 °C, within closed Petri dishes. Subsequently, these filters were examined under a microscope with a magnification range of 100–500. For contaminated filters, the particles were washed off the surface with distilled water into a 250 mL laboratory glass beaker or conical flask, followed by treatment through saline extraction and peroxide oxidation.

During the extraction and determination of microplastic concentration from sediments, we followed established procedures:

1. Drying the sample at 30 °C for a minimum of 24 h.
2. Weighing the dried sample.
3. Density separation by placing a 100-g portion of the sample in a saturated saline solution (5.75 M ZnCl solution). The volume of the solution was three times that of the sample, with an exposure time of 5–8 h and three repetitions [79–83].
4. The resulting supernatant was filtered through a 100–150 mm glass funnel using a filter for quantitative analysis (Whatman #42). The filters were replaced when clogged.
5. The filter containing retained plastic and organic matter particles was washed from the saline solution with distilled water.
6. Microplastic particles, along with organic matter from all filters related to the analysis of one sample, were washed into a glass beaker with distilled water.

7. The oxidation of organic impurities was carried out with a ratio of hydrogen peroxide to Fe(II) salt—1:1. The oxidation time was 30 min, adding 25 cm^3 of 30% hydrogen peroxide and 25 cm^3 of Fe(II) catalyst solution to a glass beaker with 150 cm^3, containing solids extracted through density separation. The beaker was placed in a water bath, with the thermostat at 50 °C, with periodic stirring for 30 min [84–86].
8. An additional portion of hydrogen peroxide was added to the beaker if undissolved organic matter was visually observed. The beaker was then covered with aluminum foil and left for a period of 8–12 h.

The density separation procedure was repeated using separation funnels, followed by filtration through a filter for quantitative analysis. The resulting filter, containing particulate matter, was placed in a Petri dish, covered with a lid, and dried at room temperature for 24 h or in a desiccator at a temperature not exceeding 35 °C [87,88]. The dried filters underwent microscopic examination, where each filter was carefully placed on a slide. The microscope was systematically navigated from edge to edge, and plastic particles were identified, categorized by type (fibers or non-fibrous materials like angular and hard fragments, flexible and thin films, or rounded and hard granules), and noted along with their respective sizes.

Qualitative analysis of microplastic particles larger than 1 mm was conducted through Fourier-transform infrared spectroscopy (FTIR) analysis. Infrared (IR) spectra were acquired using the Shimadzu IR-Prestige 21 instrument (Japan) within the wavelength range of 400–4000 cm^{-1}. The analysis was performed on the broken total internal reflection DuraSampl IR II with single reflection (prism material diamond on ZnSe substrate) from Smiths (USA). The IR spectra were matched against library databases such as IRs Polymer2, Polymer, and T-Polymer. For particles smaller than 1 mm found on filters after water filtration, they were considered plastics if they exhibited a shiny surface, bright color, sharp geometric shapes, and did not break under pressure from metal tweezers [89–91]. Depending on the amount of water filtered through the filter, the concentration of particles per 1 dm^3 was calculated. The content of microplastics in sediments was determined based on the number of particles per 1 kg of an absolutely dry sample.

Expeditions were conducted for water sampling from surface sources, organized by seasons: spring (from 19 May 2023 to 31 May 2023), summer (from 4 August 2023 to 11 August 2023), and autumn (from 14 September 2023 to 20 September 2023). The sampling locations included Kopa Lake, Zerendinskoye Lake, Borovoe Lake, and Yesil River within Astana city limits. The sampling occurred during the morning hours, specifically from 6:00 to 9:00 a.m., at each location. Water samples were obtained for the analysis of physical and chemical parameters from 8 sampling points: surface samples were collected by scooping with a metal bucket, and samples from a depth of 1.5 m were taken using a bathometer. Each sample, totaling 16 for each site in every season, was collected in 2-L containers.

For the microplastics analysis, water filtration was conducted from the surface and a depth of 1.5 m using a filtration device with polyamide filters. This process was carried out at a total of 16 filtration points for each river or lake in every season:

- With a bathometer from a depth of 1.5 m—the total volume of 50 dm^3 from each sampling point;
- From the surface with a metal bucket—volume 100 dm^3 from each point.

4. Results and Discussion

4.1. Analysis of MP Content in Water Samples

A total of 64 water samples were collected during the spring, summer, and autumn periods for the analysis of physical and chemical parameters. Water filtration was carried out at 64 points in each season. All collected samples were transported to the university laboratory for further study. When calculating microplastic concentrations, it was considered that the volume of filtered water from the surface was 100 dm^3, and from a depth of 1.5 m, it was 50 dm^3. The results of the physico-chemical analysis of water and the microplastic content in the studied lakes during spring, summer, and autumn are presented in Tables S1–S12 (Supplementary Materials). Table 11 displays the microplastic content found in sediments, and Figure 6 showcases some examples of fibers discovered in the water samples.

Table 11. Microplastic content in sediments (particles/kg).

Sediment Type	Kopa Lake	Zerendinskoye Lake	Borovoe Lake	Yesil River
Open water bottom sediments	113.48 48.32	76.51 66.16 134.45	68.62 77.15 47.41	88.11 76.10
Sediments of the water's edge zone	118.92 74.39	129.23 99.50 95.08	93.95 76.09 83.60	103.59 150.38
Splash zone deposits	212.52 147.45	152.28 105.19 147.30	84.98 85.33 99.09	121.10 120.53
Beach sediments	184.09 179.96	192.46 161.97 204.42	169.36 189.00 183.98	179.73

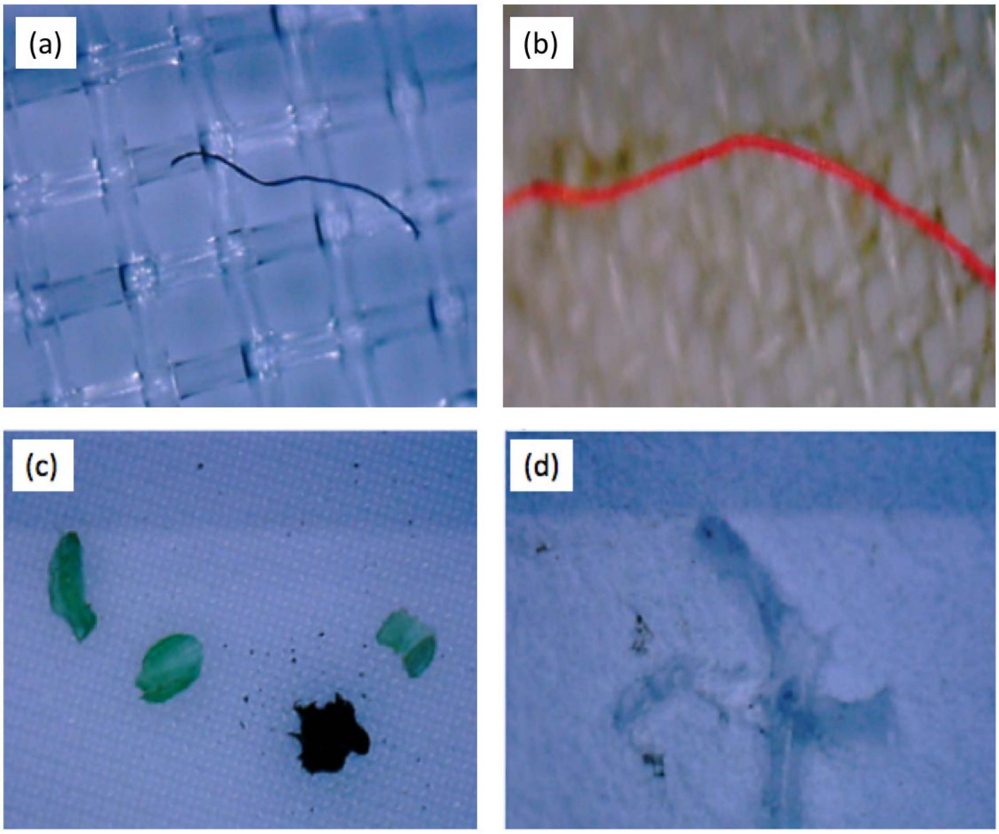

Figure 6. Samples of fibers (**a**,**b**), fragments (**c**), and films (**d**) of microplastic found in natural waters of Akmola region.

Tables 12–14 show the microplastic content found for the samples taken in Spring, Summer, and Autumn. Table 15 shows the microplastic concentration found in sediments.

Table 12. Microplastic content in surface water bodies of Akmola region (spring period).

Water Body	Fibers	Fragments	Films	Concentration (Particles/dm^3)	Average MP Concentration (Particles/dm^3)	Mean MP Concentration (Particles/dm^3) at Surface/Depth 1.5 m
Kopa Lake	120	15		1.0×10^{-2}–6.2×10^{-1}	1.4×10^{-1}	$5.5 \times 10^{-2}/2.3 \times 10^{-1}$
Zerendinskoye Lake	151	6		2.0×10^{-2}–4.4×10^{-1}	1.5×10^{-1}	$9.9 \times 10^{-2}/2.0 \times 10^{-1}$
Borovoe Lake	111	20	2	1.0×10^{-2}–4.8×10^{-1}	1.3×10^{-1}	$5.3 \times 10^{-2}/2.3 \times 10^{-1}$
Yesil River	40	30		0–4.0×10^{-1}	7.7×10^{-2}	$2.5 \times 10^{-2}/1.4 \times 10^{-1}$
Total in spring period	422	71	2	0–6.2×10^{-1}	1.2×10^{-1}	$5.8 \times 10^{-2}/2.0 \times 10^{-1}$

Table 13. Microplastic content in surface water bodies of Akmola region (summer period).

Water Body	Fibers	Fragments	Films	Concentration (Particles/dm^3)	Average MP Concentration (Particles/dm^3)	Mean MP Concentration (Particles/dm^3) at Surface/Depth 1.5 m
Kopa Lake	239	1	11	6.0×10^{-2}–4.6×10^{-1}	2.4×10^{-1}	$1.5 \times 10^{-1}/3.4 \times 10^{-1}$
Zerendinskoye Lake	440		35	1.6×10^{-1}–1.8	4.5×10^{-1}	$2.3 \times 10^{-1}/6.6 \times 10^{-1}$
Borovoe Lake	145		8	6.0×10^{-2}–2.4×10^{-1}	2.7×10^{-1}	$1.0 \times 10^{-1}/1.7 \times 10^{-1}$
Yesil River	170			6.0×10^{-2}–2.4×10^{-1}	1.5×10^{-1}	$1.3 \times 10^{-1}/1.8 \times 10^{-1}$
Total in summer period	994	1	54	6.0×10^{-2}–1.8	4.5×10^{-1}	$1.5 \times 10^{-1}/3.4 \times 10^{-1}$

Table 14. Microplastic content in surface water bodies of Akmola region (autumn period).

Water Body	Fibers	Fragments	Films	Concentration (Particles/dm^3)	Average MP Concentration (Particles/dm^3)	Mean MP Concentration (Particles/dm^3) at Surface/Depth 1.5 m
Kopa Lake	190	3		2.0×10^{-2}–1.22	1.9×10^{-1}	$5.6 \times 10^{-2}/3.3 \times 10^{-1}$
Zerendinskoye Lake	111	0		3.0×10^{-2}–3.2×10^{-1}	1.1×10^{-1}	$5.6 \times 10^{-2}/1.6 \times 10^{-1}$
Borovoe Lake	182	1		5.0×10^{-3}–4.8×10^{-1}	1.7×10^{-1}	$6.7 \times 10^{-2}/2.8 \times 10^{-1}$
Yesil River	113	1		1.0×10^{-3}–2.0×10^{-1}	1.1×10^{-1}	$4.8 \times 10^{-2}/1.7 \times 10^{-1}$
Total in autumn period	596	5		1.0×10^{-2}–1.22	1.5×10^{-1}	$5.7 \times 10^{-2}/2.4 \times 10^{-1}$

Table 15. Microplastic concentration in sediments (particles/kg).

Sampling Area	Kopa Lake	Zerendinskoye Lake	Borovoe Lake	Yesil River
Open water bottom	80.90	92.37	64.39	82.11
Water's edge zone	96.66	107.94	84.55	126.99
Splash zone	179.99	134.92	89.80	120.82
Beach	182.03	186.28	180.78	179.73

The average microplastic content in the studied samples increases from the spring to the autumn season: 1.2×10^{-1} particles/dm^3 in the spring period, 1.5×10^{-1} in summer, and 4.5×10^{-1} in the autumn sampling period. Microplastic concentrations in the lakes' water showed consistent levels, averaging 2.1×10^{-1} particles per dm^3 of filtered water, surpassing the content in the Yesil River (average for three seasons—1.1 $\times 10^{-1}$ particles/dm^3). The observed microplastic content in the natural waters of the Akmola region aligns with published data in scientific literature, ranging from 1×10^{-3} to 10 particles per dm^3 [92].

In the studied water samples, microplastics primarily manifest as fibers, constituting 93.8% of the total number of microplastic particles. Fragments follow at 3.6%, and films at 2.6%. This distribution aligns with published data, where the prevalence of fibers in surface waters ranges from 62% to 98%, fragments rank second with proportions of 2–18%, and films take the third spot with proportions ranging from 0% to 14% [93].

Despite using Whatman No. 42 filters with a pore size of 8 μm in sample processing, the lower limit for the detected microplastic particles is estimated to be 100 μm, based on the mesh size of the polyamide mesh used for water filtration. The optical particle sorting method used in filter analysis may have led to the underestimation of transparent particles. Fragment sizes ranged from 250 to 100 µm in the longest dimension, while fiber sizes along the length varied from 100 to 500 µm. The detected fibers had a diameter significantly smaller than the mesh size of the polyamide mesh (100 µm), suggesting that only those fibers caught or entangled in the mesh were detected. Consequently, it is plausible that the actual fiber content in the studied river and lakes of the Akmola region might be higher than detected.

4.2. Correlation Dependencies

For most samples, MP content showed a correlation with water turbidity and sampling depth (surface and 1.5 m depth). The average MP concentration at a depth of 5–20 cm from the surface was 8.8×10^{-2} particles/dm^3, while at a depth of 1.5 m from the surface, it was 2.6×10^{-1} particles/dm^3. Despite turbidity, no correlation was found between microplastic content and other water quality parameters. Similar results were observed for microplastic content in plankton [94]. However, a noticeable correlation between microplastic content and turbidity was identified, particularly during the spring period.

Figures 7–9 present the results obtained when comparing microplastic concentration in water samples with turbidity. Conclusions diverge when analyzing results from water samples taken at the lake shoreline (sample points one to six in every lake) compared to those observed for water samples taken at inner waters in the lakes (sample points seven and eight). Figures 7–9 have been plotted using the same scale on the X-axis (Turbidity ranging from 0 to 40 mg/dm^3) and Y-axis (microplastic concentration ranging from 0 to 1.4 particles/dm^3) to facilitate easy comparison and draw conclusions from these comparisons. Results also exhibit a significant dependence on the season, as explained below.

Figure 7 displays the turbidity–microplastic concentration correlograms for the spring, summer, and autumn seasons for the three lakes. Figure 7a presents the results for spring at points one to six, revealing highly significant correlations in all three cases. In each instance, the linear correlation coefficients R^2 exceed 0.71 for Kopa Lake, with some values surpassing 0.80 (R^2 = 0.805 for Zerendinskoye Lake and R^2 = 0.87 for Borovoe Lake).

Figure 7b illustrates the values obtained for spring at sampling points within the three lakes (points 7 and 8). Correlations remain high for Kopa Lake (R^2 = 0.63) and Zerendinskoye Lake (R^2 = 0.67), while slightly lower for Borovoe Lake (R^2 = 0.46). In all cases, MP concentrations are below 0.65 particles/dm^3, and turbidity values are below 35 mg/dm^3.

The graphs illustrating the correlations obtained for the summer season are depicted in Figure 7c,d. For points located on the lakeshores, the highest values are once again observed in Kopa Lake (R^2 = 0.85). However, in this case, MP concentrations increase significantly, with values exceeding 1.2 particles/dm^3. The values obtained for Zerendinskoye Lake are very similar to those obtained for the spring season, both in terms of the correlation coefficient (R^2 = 0.74) and turbidity and MP concentration values. In the case of Borovoe Lake (R^2 = 0.73), turbidity values recorded in summer are significantly lower than those recorded in spring (below 8 mg/dm^3), while MP concentration values remain in the same order as before, albeit slightly lower (below 0.4 particles/dm^3).

During the summer season, results for sampling points within the three lakes vary significantly compared to those observed in the spring season. No significant correlations have been detected in any of them, turbidity values are below 7.0 mg/dm^3, and MP concentration is very low, below 0.3 particles/dm^3, in all three lakes.

The correlations observed in summer, and especially in spring, do not replicate in the autumn season, as depicted in Figure 7e,f. Figure 7e displays the values obtained for points one to six in autumn for the three lakes. In none of them is the existence of any correlation observed ($R^2 < 0.13$), while turbidity values remain very low (below 7 mg/dm^3) with MP concentration values below 0.5 particles/dm^3, except for Borovoe Lake, where concentrations reach around 0.8 mg/dm^3.

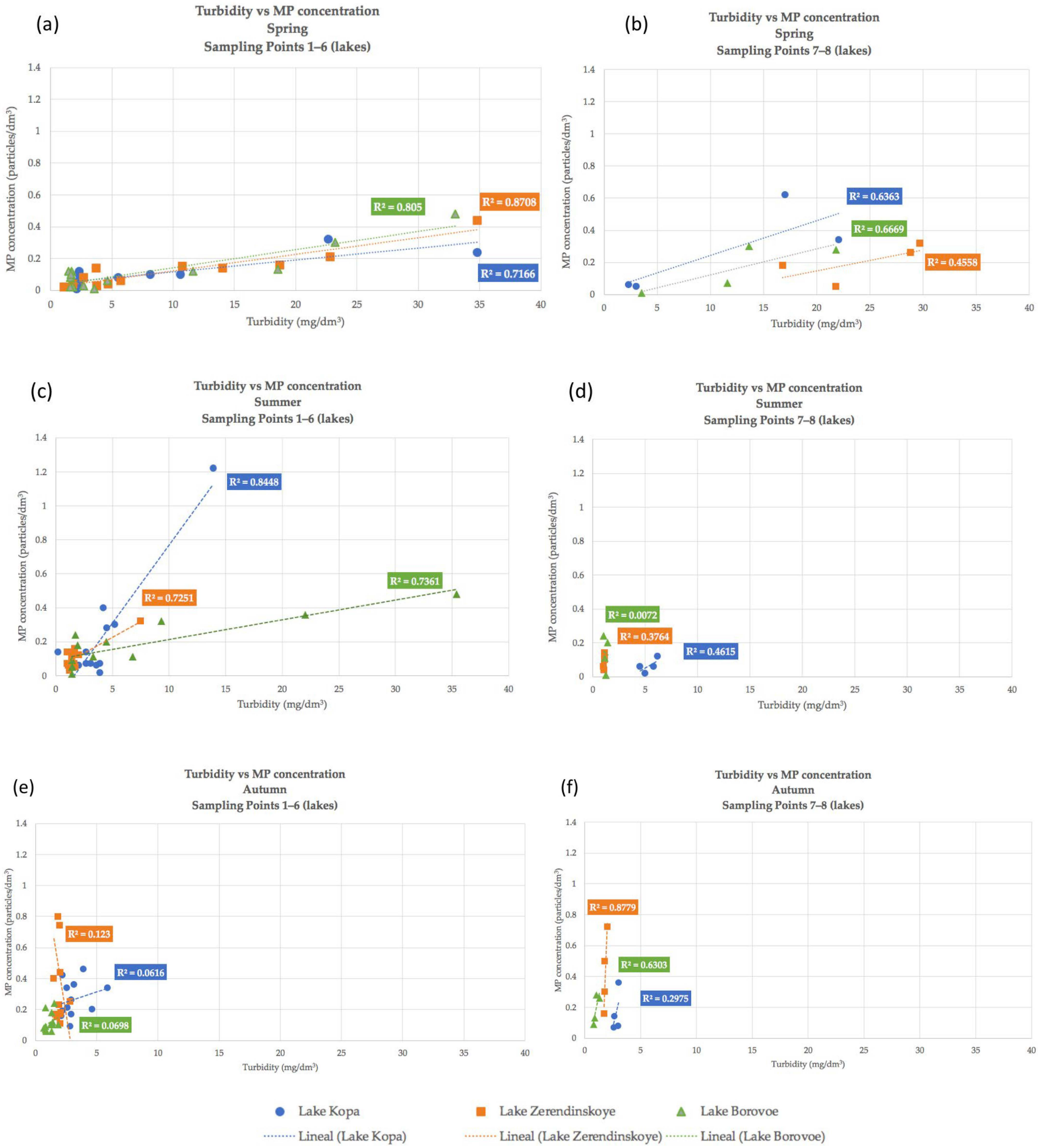

Figure 7. Turbidity vs microplastic concentration in waters for the Spring, Summer, and Autumn seasons. (**a**) Sampling points 1–6 in Spring; (**b**) Sampling points 7–8 in Spring; (**c**) Sampling points 1–6 in Summer; (**d**) Sampling points 7–8 in Summer; (**e**) Sampling points 1–6 in Autumn; (**f**) Sampling points 7–8 in Autumn.

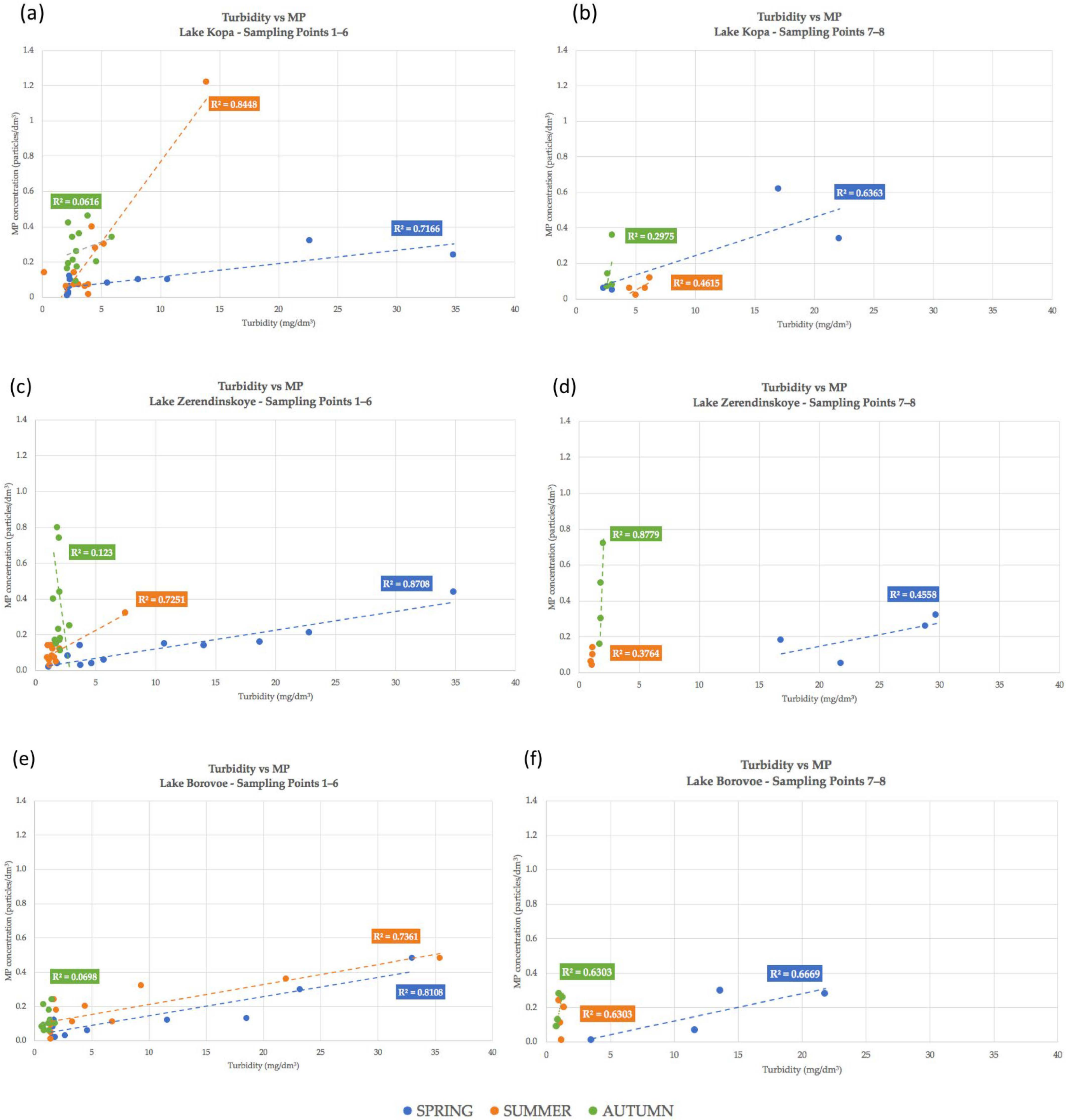

Figure 8. Turbidity vs microplastic concentration in waters: (**a**) Kopa Lake samples 1–6; (**b**) Kopa Lake samples 7–8; (**c**) Zerendinskoye Lake samples 1–6; (**d**) Zerendinskoye Lake samples 7–8; (**e**) Borovoe Lake samples 1–6; and (**f**) Borovoe Lake samples 7–8.

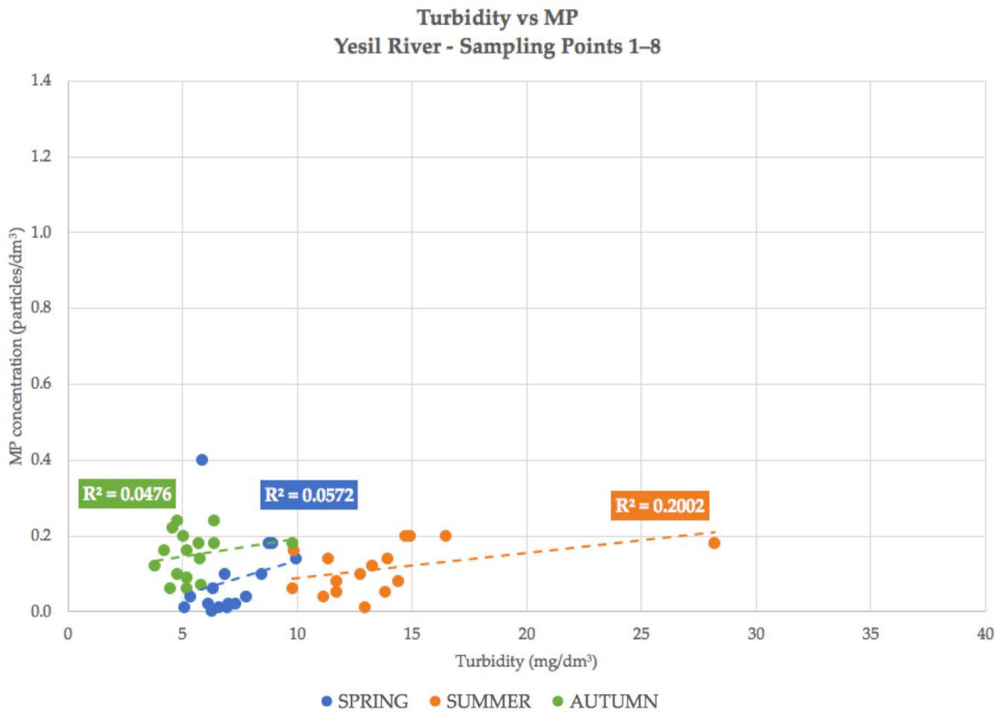

Figure 9. Turbidity vs. microplastic concentration in the Yesil River waters.

For the autumn season, values observed within the lakes (points seven and eight) are very similar to the corresponding values obtained for points located on the shores. In this case, turbidity values do not exceed 5 mg/dm^3 in any of the analyzed cases, and MP concentration values are below 0.4 particles/dm^3 for Kopa Lake and Zerendinskoye Lake, while for Borovoe Lake, they are slightly higher, exceeding 0.7 particles/dm^3. For sampling points seven and eight, correlation coefficient values exceeding 0.60 have been found for Zerendinskoye Lake (R^2 = 0.63) and Borovoe Lake (R^2 = 0.88).

Figure 8 shows the relationship between turbidity and microplastic concentration in waters for three lakes during the spring, summer, and autumn seasons. Figure 8 has been generated to observe the temporal evolution of turbidity and MP concentration more clearly in each lake independently.

Figure 8a,b illustrate the results obtained for Kopa Lake. For sampling points located on the shores of Kopa Lake, the results depicted in Figure 8a highlight strong correlations between turbidity and MP concentration in Kopa Lake during the spring (R^2 = 0.72) and summer (R^2 = 0.85) seasons. The highest turbidity values were observed in spring, reaching values close to 35 mg/dm^3. The highest MP concentration values in Kopa Lake were recorded in the summer season, with concentrations exceeding 1.2 particles/dm^3. However, during the autumn season, these correlations disappear, while turbidity and MP concentration values decrease significantly. During the autumn season, turbidity values in Kopa Lake are below 7 mg/dm^3, and MP concentration does not exceed 0.5 particles/dm^3.

Figure 8b displays the results obtained for sampling points within Kopa Lake (points seven and eight). The high correlations observed for the spring and summer seasons at points one and six are now only preserved for the spring season (R^2 = 0.63), with a drastic decrease in the correlation coefficient in the summer (R^2 = 0.46) and autumn (R^2 = 0.30) seasons. The highest turbidity values are still observed in spring, although they decrease to approximately 22 mg/dm^3. The highest MP concentrations are also observed in spring, reaching slightly above 0.6 particles/dm^3. It is noteworthy that compared to the values observed at points 1–6 in summer, MP concentrations in summer at the interior sampling points in Kopa Lake only have values of 0.2 particles/dm^3.

The results obtained for Zerendinskoye Lake are presented in Figure 8c,d. For sampling points located on the shores of Zerendinskoye Lake, the results depicted in Figure 8c highlight a very strong correlation between turbidity and MP concentration during the spring (R^2 = 0.87). However, the correlation decreases in the summer (R^2 = 0.72) and autumn (R^2 = 0.12) seasons. The highest turbidity values were observed in spring, reaching values close to 35 mg/dm^3. Surprisingly, the highest MP concentration values in Zerendinskoye Lake were recorded in the autumn season, with concentrations exceeding 0.8 particles/dm^3. However, during the autumn season, turbidity values are lower than 3 mg/dm^3, and the correlations found for spring and summer disappear. During the spring season, turbidity values in Zerendinskoye Lake are the highest (just below 35 mg/dm^3), and observed MP concentrations are just over 0.4 particles/ dm^3.

Similarly, the results obtained for Borovoe Lake are shown in Figure 8e,f. As with Kopa Lake, the highest correlations were obtained for the spring (R^2 = 0.81) and summer (R^2 = 0.73) seasons. The maximum turbidity values are very similar in these cases (35 mg/dm^3 for summer and 33 mg/dm^3 for spring), and similar MP concentrations are observed, with maximum values around 0.5 particles/dm^3. Similar to what was found in Kopa Lake and Zerendinskoye Lake, the highest MP concentrations were found in the summer season, although their values are very similar to those observed in spring. As mentioned for Kopa Lake and Zerendinskoye Lake, the correlations found during spring and summer disappear in the autumn season (R^2 = 0.07), where turbidity values decrease drastically to below 3 mg/dm^3, and MP concentrations are below 0.25 particles/dm^3.

The conclusions already presented for Kopa Lake and Zerendinskoye Lake are valid for the sampling points located within Borovoe Lake. In this case, the regression coefficient value in spring for sampling points seven and eight reaches the value R^2 = 0.67, a slightly higher value than that recorded for Kopa Lake (R^2 = 0.63) but lower than that recorded for Zerendinskoye Lake (R^2 = 0.87).

Figure 9 displays the results obtained for the Yesil River. A discernible correlation between turbidity and MP concentration in the Yesil River water has been identified only during the summer season, with a very low correlation coefficient (R^2 = 0.20). The maximum values of turbidity are observed in summer (28 mg/dm^3). However, the highest values for MP concentration are found in the spring season (0.4 particles/dm^3).

A discernible pattern emerged from the analysis, indicating that samples taken from a depth of 1.5 m exhibit a higher microplastic content compared to water samples taken and filtered from the surface of the reservoir at the same point.

4.3. Analysis of MP Content in Sediment Samples

The results of sediment analysis (including bottom sediments, water edge, splash zone, and beach sediments located perpendicularly to each other) revealed a decrease in microplastic concentrations from the coastal zone towards the sediments in open waters. The average concentrations of microplastics were as follows: 79.63 particles/kg in bottom sediments, 102.47 particles/kg in the water edge zone, and 127.58 particles/kg in the splash zone. The beach sediment zone exhibited the highest content of microplastics, with an average of 179.73 particles/kg. In terms of particle shape, microplastic fibers predominated in sediments (69.6%), followed by fragments (19.1%), with films and granules accounting for 11.3%.

Published data suggest that predominant particles may include fibers (up to 77%) [95] and fragments (up to 73%) [96]. However, in most studies, fibers are identified as the predominant microplastic particles [97]. The size of microplastic fragments and films in water ranged from 250–100 µm in the largest dimension, while the size of fibers along the length ranged from 100 to 500 µm, consistent with published data [98]. Particles larger than 500 µm were found in beach sediments, constituting an average of 40.5%.

4.4. Analysis of FTIR Results

The type of microplastic was determined by comparing the IR spectra of the samples with those in libraries (IRs Polymer2, Polymer, T-Polymer, T-Organic) and by analyzing absorption bands caused by stretching and bending vibrations of groups characteristic of certain types of polymers [99]. Based on the results of FTIR analysis, 95% of the samples were identified as particles of polymer origin (see Figure 10). The IR spectra of 3% of the samples were identified as belonging to the natural polymer cellulose, while unidentified structures accounted for 2% of microplastics (likely due to the degradation of the structures in the natural environment).

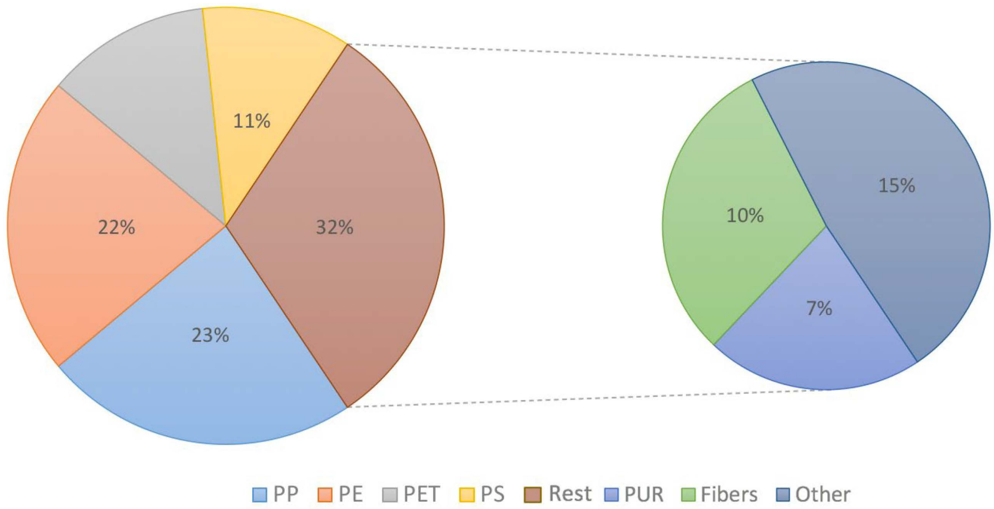

Figure 10. FTIR analysis. Qualitative composition of microplastics.

The total amount of identified microplastics is predominantly comprised of particles of polypropylene (23.3%), polyethylene (22.2%), polyethylene terephthalate (12.2%), and polystyrene (11.1%). Synthetic fibers (such as polyacrylic, polyamide, and polyester fibers) account for 9.5% of all identified microplastic particles. Similar results, indicating the predominance of microparticles of polypropylene and polyethylene in natural water bodies, are explained by their resistance to degradation and the challenges posed to decomposition by microbial biota [100]. Consequently, the inadequate management of plastic waste in Kazakhstan contributes to the presence of microplastics in all natural water bodies and their sediments in the Akmola region [19,101].

Based on the identified qualitative composition of microplastics, we can infer that the primary sources in water bodies and sediments of the Akmola region in Kazakhstan are likely household plastic waste, particularly disposable tableware made from polypropylene, polyethylene terephthalate, and polystyrene, along with packaging materials predominantly composed of polypropylene, which are widely utilized in Kazakhstan [102,103]. Another significant source of microplastics entering natural water bodies could be domestic wastewater, which carries rinsing water from washing activities, containing synthetic fibers such as polyacrylic, polyester, and polyamide fibers [44,104]. Additionally, storm drains from areas such as car repair shops and roadways may contribute to microplastic pollution, as they can carry rubber waste, including polyurethanes [105].

5. Conclusions

This paper presents, for the first time in Kazakhstan, the results of a comprehensive sampling campaign assessing microplastic (MP) content in three lakes and one river, alongside various water quality indicators. The investigation into MP concentrations revealed distinct seasonal and locational patterns. Strong correlations between turbidity and MP concentration were observed in Kopa Lake, Zerendinskoye Lake, and Borovoe Lake during spring and summer, with peak values in these seasons. However, these correlations diminished in autumn, with consistently higher MP concentrations during summer. In the Yesil River, a discernible correlation between turbidity and MP concentration emerged only in summer, with a low correlation coefficient.

The analysis extended to sediment samples, exploring different zones including bottom sediments, water edge, splash zone, and beach sediments. Results indicated a decreasing trend in microplastic concentrations from the coastal zone towards the sediments in open waters. The beach sediment zone exhibited the highest MP content, with microplastic fibers being the predominant particle type. Particle size analysis revealed variations, with microplastic fragments and films in water ranging from 250–100 µm, while fibers ranged from 100 to 500 µm. Larger particles exceeding 500 µm were notably found in beach sediments, constituting an average of 40.5%.

Overall, the findings highlight the complexity of MP distribution, influenced by seasonal variations, river or lake characteristics, and sediment composition, providing valuable insights for understanding and managing microplastic pollution in aquatic environments.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/w16071051/s1, Tables S1–S12: Results of the studies of water quality at Lakes Kopa, Zerendinskoye, and Borovoe and at Yesil River. The Excel file with the details of the correlation analysis is also provided.

Author Contributions: Conceptualization, N.S.S. and J.R.-I.; methodology, N.S.S.; software, J.R.-I. and M.-E.R.-C.; validation, N.S.S.; formal analysis, N.S.S. and J.R.-I.; investigation, N.S.S. and J.R.-I.; resources, N.S.S. and J.R.-I.; data curation, N.S.S., L.A.M., Z.O.T. and A.D.M.; writing—original draft preparation, N.S.S. and J.R.-I.; writing—review and editing, J.R.-I. and M.-E.R.-C.; visualization, J.R.-I. and M.-E.R.-C.; supervision, N.S.S. and J.R.-I.; project administration, N.S.S.; funding acquisition, N.S.S. and J.R.-I. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data is contained within the article or Supplementary Materials.

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