

Article

Microplastic Pollution in the Surface Waters from Plain and Mountainous Lakes in Siberia, Russia

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Abstract: Microplastics (MPs) contaminations of freshwater and marine environments has become a global issue. Lakes in southern Siberia provide a wide range of ecosystem services and are essential elements in the annual and interannual runoff distribution of the Great Siberian Rivers. However, the extent of their MPs pollution remains unknown. In this paper, for the first time, we analyze the concentrations, composition, and spatial distribution of MPs in six lakes in southern Siberia. The studied lakes are located both in the Altai mountains and the West Siberian plain. Some of them are significantly impacted by human activities, while others are located in protected areas with no permanent population. Nevertheless, MPs were detected in all of the studied lakes. MPs concentrations ranged from 4 to 26 MPs L⁻¹. Comparing with other inland lakes, South Siberian lakes presented moderate MPs concentrations. Among the registered MPs forms, fragments and films were dominant, with a size range between 31 and 60 nm. The MPs' sources depend on local human activities (fishing, transport, landfilling). Therefore, sufficiently high concentrations were observed even in remote lakes. The present study set a baseline that emphasizes the need for increased attention to waste management and sustainable water use in Siberian freshwater environments.

Keywords: microplastic; SEM/EDS; surface waters; lakes; Altai Mountains; West Siberian Plain



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1. Introduction

Plastic pollution occurs in most environments worldwide [1]. As of 2015, about 79% of all plastic globally produced ends up in landfills or natural environments [2]. Due to the material properties, plastics hardly decompose and, thus, remain for a long time in the environment. The widespread use of plastics results in a large variety of litter sizes from meter to nanometer range. Plastic debris less than 5 mm is usually considered as microplastics (MP) (see review by [3]). However, much smaller particles are also subdivided into nanoplastics (NP), which have diameters smaller than 1 µm [4].

Recently, many efforts have been devoted to study the pollution of microplastics in aquatic environments [5,6]. Despite the fact there is a lot of research describing microplastics occurrence and accumulation in marine environments, it is still limited for freshwater systems (see recent reviews by [7,8]). Considered as semi-closed systems with varying hydrographic conditions, freshwater lakes may suffer even more than ocean and coastal regions from the presence of microplastics [9]. Microplastics found in freshwater ecosystems can contaminate aquatic biota, cause physical damage, and transfer into the food chain [1,10,11], while toxic effects are assumed to be low [12]. Since the emergence of systematic studies, microplastic pollution has been identified in lakes of different dimensions in North America [13,14], South America [15], Europe [9,16], Asia [17–19], and Africa [20]. Despite growing evidence, the terrestrial component of the global microplastic budget is not well understood because sources, stores, and fluxes are poorly quantified [21,22].

Major pathways carrying microplastics to freshwater environments have been identified as effluent [23–25], runoff from urbanized areas [14,26,27], degradation of plastic waste in water bodies [28], and atmospheric deposition [29]. While fluvial processes must play a key role in microplastic transfer, lakes may act as a buffer store of a not-yet-identified temporal dimension for microplastic accumulation [16].

Lakes located in remote and low populated areas in Asia can suffer from microplastics pollution without proper waste management [17,18]. In Russia, where serious problems with waste disposal occur with low recycling rates and primary landfilling [30,31], research on microplastic pollution has just begun. In Lake Baikal, high levels of microplastic pollution have been identified in the water near tourist areas and coastal settlements [32]. Significant concentrations of microplastics in bottom sediments have been found within Onega, one of Europe's largest lakes, even higher than in the Baltic Sea [33]. Runoff of the Great Siberian Rivers contributes significantly to the microplastics pollution of the Arctic Seas [34]. This study has also shown that marine-borne microplastics (microplastics advected from the North Atlantic), and river-borne microplastics have distinctly different physical (size, morphology, weight) and chemical (polymer type) characteristics. However, only one study on the morphology, composition, and concentrations of microplastics in Siberian rivers [35] does not allow for tracing pollution flows. Therefore, despite a growing number of studies, many regions of Northern Eurasia lack preliminary baseline data.

Currently, there are almost no data on the microplastic contamination of remote lakes with no permanent population in their catchments [8]. Microplastics can come with atmospheric fallout even in remote protected areas [36]. There are numerous lakes of different dimensions in Siberia both directly exposed to human impact and almost completely undisturbed. In this paper, we analyze, for the first time, microplastic pollution in six inland lakes in the Altai Mountains and the southern West Siberian Plain. The present study focuses on the concentration, distribution, and characteristics of microplastics, addressing their identification, source evaluation, and correlation with lake and catchment features. Three of the studied lakes are located in almost pristine areas, partially or entirely in strictly protected areas where all activity is prohibited. At the same time, three other lakes are significantly impacted by anthropogenic activities, such as livestock, agriculture, mineral extraction, landfilling, and tourism. Freshwater ecosystems in southern Siberia provide many ecosystem services, including water for human consumption and the development of economic activities and serve as critical habitats for various species. Among other issues, collecting baseline data for microplastics in southern Siberia is crucial for understanding microplastics flows in northern Eurasia as, while some of the studied lakes are located at the Great Siberian Rivers' headwaters, others belong to endorheic basins.

2. Materials and Methods

2.1. Study Area

The six lakes studied are Talmen Lake (TY), Dzhulukul Lake (DZ), Teletskoye Lake (TL), Zludyri Lake (ZL), Degtyarka Lake (DK), and Kuchuk Lake (KH) (Figure 1). The investigated lakes are located in the southern part of Western Siberia in Russia. This region is the most densely populated part of Siberia, although it includes many pristine areas. The lakes are classified as mountainous, located within the Altai Mountains (TY, DZ, TL), and plain lakes, localized in the far southern part of the West Siberian Plain (ZL, DK, KH). They differ in size, origin, and climate. A wide range of available morphometric and geographic features were summarized in Table 1 [37,38]. Plain lakes are located in populated areas and are mainly used for local fishing, recreation, watering places for livestock, and mining. Mountainous lakes are situated in sparsely populated areas and mainly used as tourist destinations.

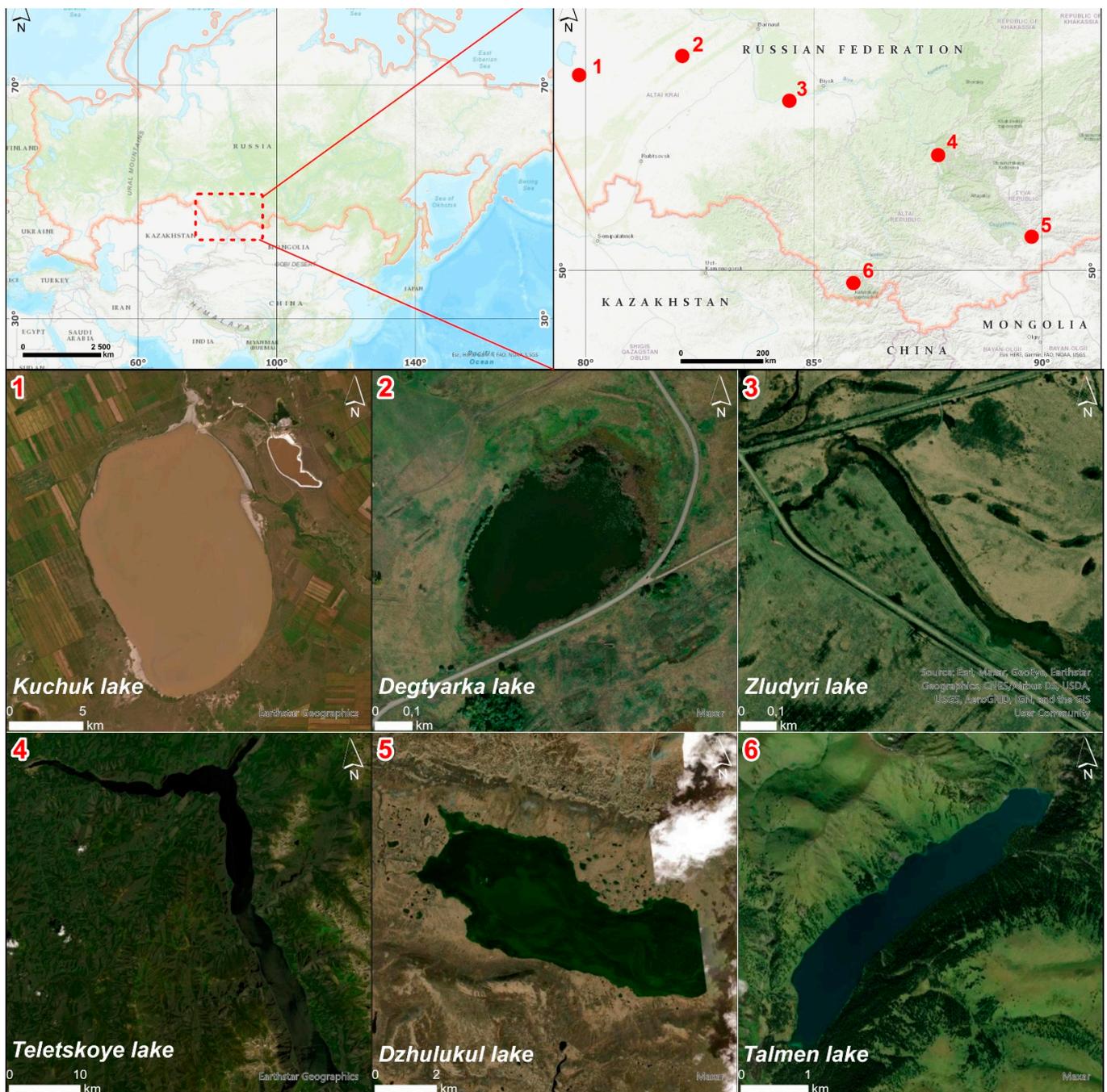


Figure 1. Map of the study area location within Russia and localization of the studied lakes. (1) Kuchuk Lake (KH). (2) Degtyarka Lake (DK). (3) Zludyri Lake (ZL). (4) Teletskoye Lake (TL). (5) Dzhulukul Lake (DZ). (6) Talmen Lake (TY).

All mountainous lakes are fully or partially located in strictly protected areas (Katunsky and Altaisky State Nature Reserves). DZ and TY present no permanent population in their surroundings. TY is a large ribbon lake in the headwater of the Katun river basin. TY is fed by watercourses flowing down from the Katun Range (also after glacier melt). DZ is a high-mountain lake located in a vast hollow, from which the Chulyshman River flows into TL. TL is one of the biggest and deepest mountain lakes in southern Siberia. It is a tectonic lake, long and open (the Biya river outflow). More than 70% of the lake's water inflows are provided by the Chulyshman River [39]. The main population is located in the north-western lakeshore. Motorboat traffic is allowed on TL and TY. All mountainous

lakes are essential elements in the hydrological functioning of the Katun and Biya rivers, which form the Ob River after their confluence.

Table 1. Main morphological and geographical characteristics of each lake and primary watershed information obtained from the literature and official statistics.

Lakes	Lake Altitude (m)	Area (km ²)	Maximum Depth (m)	Watershed Area (km ²)	Watershed Position	Inflow	Outflow	Population within a Watershed (n)	Distance to a Settlement (km)
Talmen (TY) 49°49′12.9″ N 85°49′06.2″ E	1531	3.9	68	117	Header	Yes	Yes	0	30
Dzhulukul (DZ) 50°29′31.6″ N 89°41′25.4″ E	2199	30	7	NA	Header	Yes	Yes	0	50
Teletskoye (TL) 51°33′14.8″ N 87°40′39.0″ E	434	223	325	19,500	Intermediate	Yes	Yes	11,624	0
Zludyri (ZL) 52°21′30.4″ N 84°23′52.4″ E	153	NA	NA	NA	Terminal	No	No	3159	0.5
Degtyarka (DK) 52°21′30.4″ N 84°23′52.4″ E	220	NA	NA	NA	Terminal	No	No	784	1.5
Kuchuk (KH) 52°41′56.9″ N 79°46′50.4″ E	98	181	3,3	3240	Terminal	Yes	No	19,917	1.5

Plain lakes have almost no outflow, and water loss is mostly by evaporation. ZL is an oxbow lake on a broad Ob's floodplain near the river's outlet from the Altai Mountains to the West Siberian Plain. ZL has a connection to the river only in years with severe floods (once every 15–20 years). DK is situated on the Ob plateau within an extended ancient flow depression [40]. The water level in the lake fluctuates greatly during the season. DK has in close proximity the A-321 federal highway, Podstepnoe village, and several farms. KH is a large, closed lake on the Kulunda Plain, which is the only saline lake among those surveyed. It belongs to a vast endorheic basin on the Ob-Irtysh interfluvium. Stepnoye Ozero town and a mirabilite mining complex are located on the lakeshore of KH. Kulunda is a huge agricultural region that suffered from a lack of precipitation and desertification [41].

2.2. Sampling Collection and Quality Control

We carried out sampling during summer 2020. The water samples were collected at eight sampling points. Therefore, five lakes contained one representative sampling point each. In the much larger TL, we sampled at three pelagic sampling points. The first point, TL(U), was located in the southern part of the lake near the Chulyshman River, which is the major inflow. The second point, TL(M), was in the central part of the lake (near Yailu village). The third sampling point, TL(L), was in the northern part near the Biya River outflow (see the map of sampling points).

During sampling and further analysis, we followed the following several measures: (1) use only glass and metal equipment; (2) clean the surfaces with 90% ethanol and paper towels, wash the equipment with ultrapure water; (3) use expendable materials directly from packaging; (4) use procedural blanks to control; (5) keep samples covered as much as possible and handle them in a fume hood or by covering the equipment during handling.

We took surface water samples from the depth of 30 cm into prepared 5-liter glass jars. These glass jars were preliminarily washed in the laboratory with 90% ethanol and then three times with ultrapure water. The ultrapure water was controlled for the presence of microplastic particles, as the sampling glass jars, i.e., the blank control was fully implemented. After sampling, samples were hermetically sealed and additionally covered with aluminum foil to prevent secondary contamination. When cooled, samples

were taken to the laboratory, where they were filtered using a vacuum filter pump with metal funnels (model PVF 35/1). The contact parts (with the samples) were metal-only and pre-washed with 90% ethanol and ultrapure water. Samples were filtered through glass microfiber filters Whatman GF/A, then dried in Petri dishes, and stored until analysis in labeled paper boxes.

2.3. Laboratory and Statistical Analysis

We used scanning electron microscopy plus energy-dispersive X-ray spectroscopy (SEM/EDS) analysis on the glass microfiber filters through which water samples were filtered. SEM/EDS allowed many microplastic particles to be screened in a relatively short time. SEM/EDS screening utilized surface morphology and elemental composition to determine whether each particle was potentially a plastic. First, all samples were mounted on aluminum SEM stubs. Then, we covered samples with conduction layers by sputtering Au-Pd non-conducting microplastics scraps. SEM/EDS analyses were conducted using an SEM S-3400N (Hitachi Science Systems Ltd., Narashino-shi, Japan) with magnification up to 300,000, resolution up to 3.0 nm, and dispersion X-ray analysis system QUANTAX EDS SDD detector XFlash 4010 (Bruker AXS Microanalysis GmbH, Berlin, Germany). We examined the samples under 200 \times magnification in the first stage, which allowed us to identify large particles (several hundred nm). Then, the same samples were analyzed under 400 \times magnification, which provided images of particles down to a few nm. SEM/EDS provided high-resolution imaging of particle surface structures, as well as elemental composition signatures. This technique is very useful for the recognition of organic (rich in Ca/Mg/Sr) and inorganic (minerals, salts) and microplastics (rich in C/Cl/S/Ti) [42]. Therefore, EDS was used to screening for likely microplastics and exclusion non-plastics.

Each microplastic particle was classified into five categories: fibers, films, fragments, foams, and pellets. Additionally, we determined the length along the long axis (nm). There are the following three microplastic size classes, which reflect current sampling and processing practices: $1 \leq 100$ nm, $100 \leq 350$ nm, and $350 \text{ nm} \leq 5$ mm [43]. Our research focused on the first two classes, including the smallest particles, as it is most challenging to obtain results on such a small class. Difficulties frequently arise with trawling using nets, which cannot capture small particles (mesh size 300 nm). Despite this limitation, it remains the basic method in most studies. However, >95% of particles in surveyed products were smaller than the 300-nanometer minimum diameter [44].

The abundance unit of microplastics in all samples is the number of microplastics per liter (MPs L⁻¹; [45]). The average values and standard deviation (SD) were calculated using Microsoft Office Excel 2018. Two-sided Fisher exact tests were used to determine ($\alpha = 0.05$) if the shape and size proportion varied between the studied lakes. Statistical analysis and all charts were performed using R [46].

3. Results and Discussion

3.1. Abundance and Distribution of the Microplastic

Microplastics were found in all the lakes studied. The abundance of microplastics varied from 4 to 26 MPs L⁻¹, with an average abundance of 11 ± 7 MPs L⁻¹ in the surface water (Figure 2). For the correct comparison of microplastics concentrations, it is necessary to consider the following several conditions: the type and location of the study object (reservoir or watercourse), and the sampling methods (nets, pump, or sampler/jar/bucket). It is important to note that the use of buckets, jars, and samplers involves filtration in the laboratory, and filtration conditions in the laboratory would be better controlled. The following physical and chemical methods for microplastics characterization are also worth considering: Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, Gas chromatography coupled with mass spectrometry detector or SEM-EDS [42]. All the mentioned parameters and conditions largely determine the qualitative and quantitative characteristics of microplastics in water bodies.



Figure 2. Microplastics concentrations (MPs L⁻¹).

Compared with the other lakes worldwide, inland Siberian lakes have a moderate microplastics concentration, considering similar sampling methods (no nets or pumps). Therefore, using data for 98 lakes worldwide [8], we could compare our data with only a few studies, mainly in Asia. For example, the reported concentration of microplastics in Taihu Lake (China) ranges from 3.4 to 25.8 MPs L⁻¹ [47] and in Poyang Lake from 5 to 34 MPs L⁻¹ [48]. Other lakes and streams in China (Chengdu Plain) showed 6.11–44.08 MPs L⁻¹ in the water with an average abundance of 15.88 ± 3.13 MPs L⁻¹ [12]. In Wuliangshuai lake water, the microplastic concentrations ranged from 3.12 to 11.25 MPs L⁻¹ [49]. These concentrations were slightly higher than in some other lakes, for example, in the high-mountain lake in the Carnic Alps, where microplastics were determined as one item in 3 L (0.33 MPs L⁻¹). However, in this study, water sampling was performed using the “Apstein” plankton net (0.30 m in diameter, 0.90 m in length, and 50 μ m mesh size), and analysis was conducted using Fourier transform infrared spectroscopy equipped with an MCT-A detector [50]. Lower microplastic concentrations were also obtained in Yellowstone Lake in North America (2–11 MPs overall). Sampling was performed using a plankton net (Wildco[®] mesh size 80 μ m, diameter 20.32 cm, net material Nitex[®] nylon), and samples were analyzed using FTIR and Raman spectroscopy [51]. Therefore, comparing our data on microplastic concentrations in lake waters with results obtained with similar methods showed a high consistency.

We observed no essential differences in the microplastic concentrations in the mountainous and plain water bodies. The concentrations appeared to be dependent on the nearby surroundings and local human activities. The highest microplastic abundance occurred at DK and reached 26 MPs L⁻¹. Such a high concentration was expected given the endorheic type of the lake and the highway, the settlement, and the farms near the DK shoreline. However, similar in dimension and surroundings, ZL showed low concentrations (4 MPs L⁻¹). The surrounding area is also occupied by pasture and actively used by the local population. Therefore, it is expected that local sources of microplastics inputs and their configuration play a dominant role in the concentrations. DZ, located within the

Altaysky Nature Reserve, was expected to show low concentrations of microplastics (about 5 MPs L⁻¹). Anthropogenic loads within the lake basin are minimal; therefore, microplastic particles likely came with atmospheric fallout. More frequented by tourists, TY showed higher values of microplastics concentrations (8 MPs L⁻¹). Potential sources of input could be tourist litter, motorboat traffic, and atmospheric transboundary transport. The industrially developed regions of Kazakhstan are located 100–150 km west of TY. Considering the lack of permanent population and the outflow, TY concentrations can be assessed as relatively high. The concentrations of microplastics in KH reached a similar 8 MPs L⁻¹. At the same time, KH has a significantly more extensive and populated catchment and is also endorheic. Most likely, agricultural activities in the basin and lack of direct input sources of MPs from settlements formed moderate concentrations of microplastics.

In TL, concentrations varied from inflow to outflow in the southern and northern parts (Figure 2). The maximum concentration of microplastics (18 MPs L⁻¹) was detected in the southern part of the lake, near the inflow of the largest tributary—Chulyshman river. In the populated northern and the central parts, concentrations did not exceed 9 MPs L⁻¹. The lower reaches of the Chulyshman River have a significant intensity of tourist activity and permanent population. This activity determined a significant contrast between the source of the river in DZ and the estuary in TL. In addition, the outflow in the northern part may have contributed to the rapid transfer of microplastics into the Biya River.

3.2. Shape and Size of the Microplastic

All categories of microplastic forms were detected in the studied lakes. For all the lakes, the ratio was as follows: films—21%, fragments—37%, fibers—9%, foam—14%, and pellets—19%. In contrast to studies where fibers and fragments were most often found [52], the number of fibers was quite low. However, fragments were the dominant form of microplastic.

The differences of the microplastic forms across the lakes were statistically significant (Fisher test p -value = 0.01682). The distribution was irregular and related to the types of human activities (Figure 3). As noted recently, human activities such as washing in river water and fishing with broken nets directly lead to an increase in fibers [12]. Absolutely all of the particles found in ZL were films. Locals use this oxbow lake for fishing and washing cars. A large proportion of films (67%) was also observed in the northern part of TL near the village of Artybash, with the highest density of vehicles and water transport. The maximum number of fragments was found in the southern part of TL (64%), DZ (50%), and DK (43%). The fragments of microplastic coatings are generated during the rain scouring process of road surfaces [53]. In turn, DK is adjacent to the highway. The Chulyshman river delivers MPs from the watershed to the southern part of TL. Fibers were found in KH (50%) and the central part of TL (40%). Microplastic fibers are known to be released from textiles due to wear and tear and washing [54]. In central TL, locals use the lake to rinse laundry, which may presumably be responsible for the fiber dominance. The reasons for high fibers concentrations in KH are not entirely clear. In addition, no pellets (i.e., primary microplastics) were observed in the lakes with the lowest concentrations of microplastics (ZL and DZ).

Microplastic particle sizes varied from 10 to 960 nm. This range is comparable with lakes in Finland, where the particles were 20–300 nm in size [55]. The total number of particles larger than 300 nm did not exceed 15%. In this regard, our primary attention was focused on smaller particles. Particles 31–60 nm were the most common. They were present in almost all the lakes (Figure 4). This result corresponds well with published data on microplastics size in the world's lakes, which showed that microplastic particles were predominantly less than 1 mm, and even < 500 nm [8]. The differences of microplastic size classes across the lakes were also statistically significant (Fisher test p -value = 0.003257). The lakes with the highest concentrations of microplastics also showed the highest diversity of size groups.

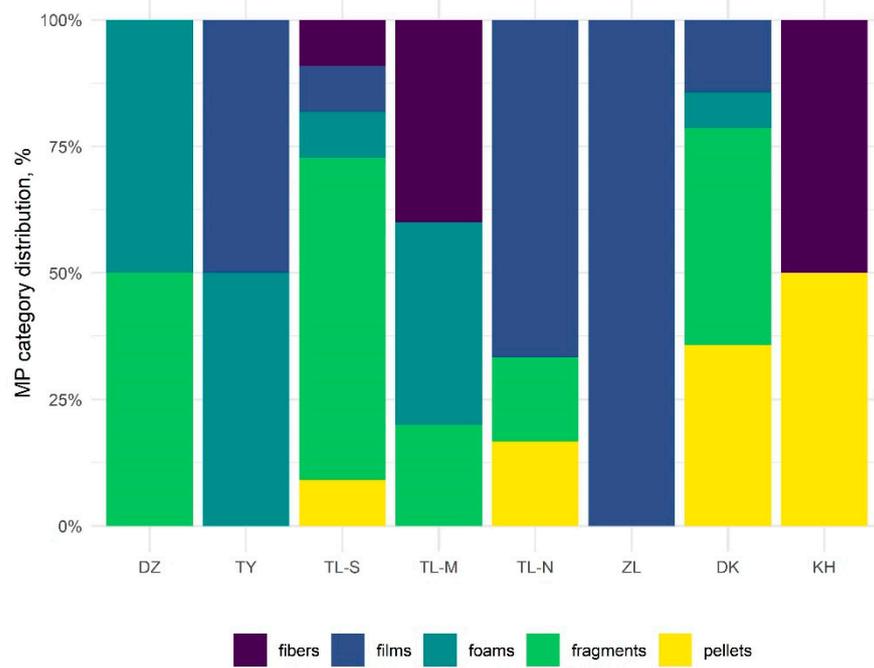


Figure 3. Microplastics category distribution (%).

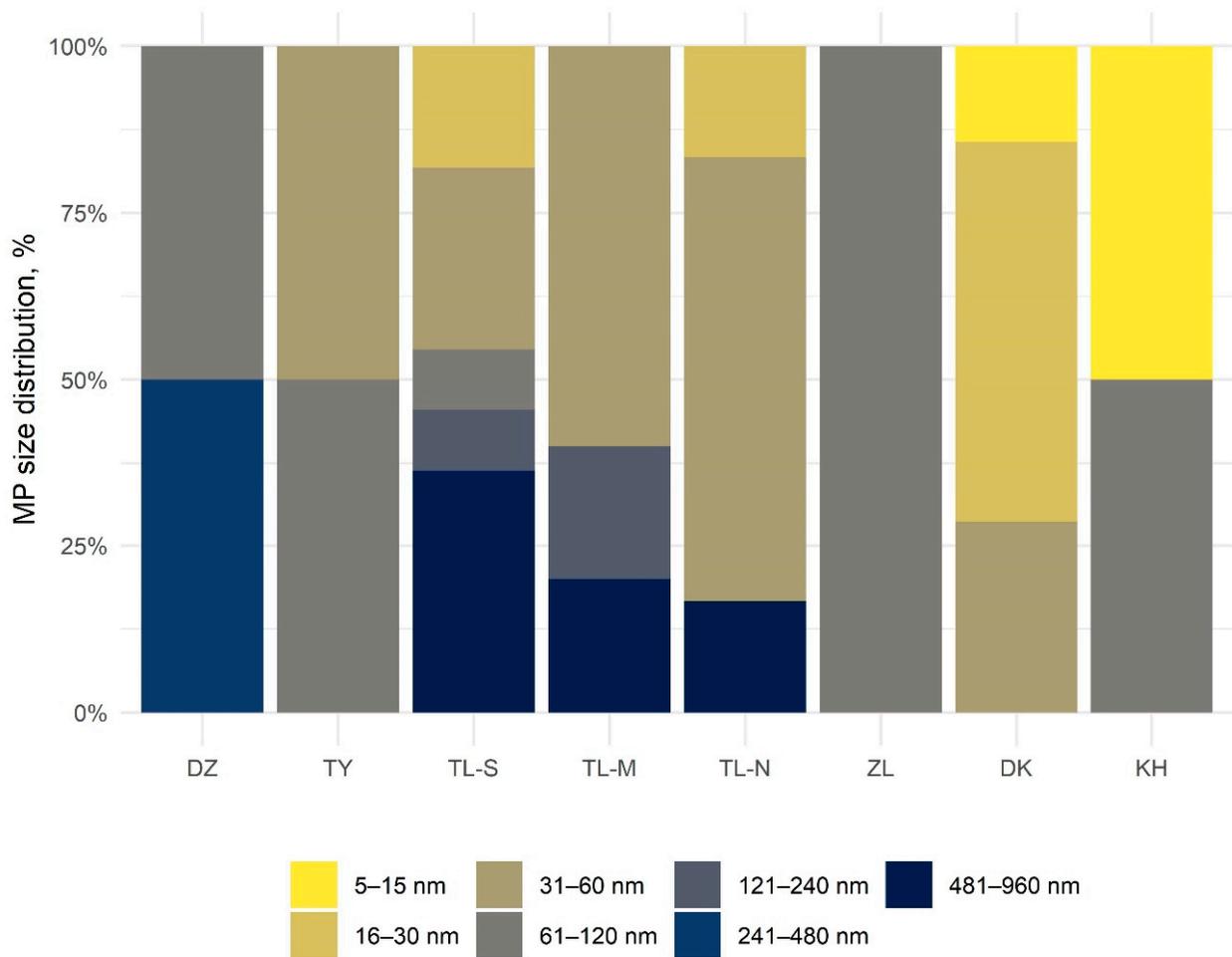


Figure 4. Microplastics size distribution (%).

Fragments and films were represented in almost all size groups (Figure 5). However, we found no statistically significant differences in the belonging of microplastic forms to individual size classes (Fisher test p -value = 0.08069). In other words, particles of any shape could have the most different sizes.

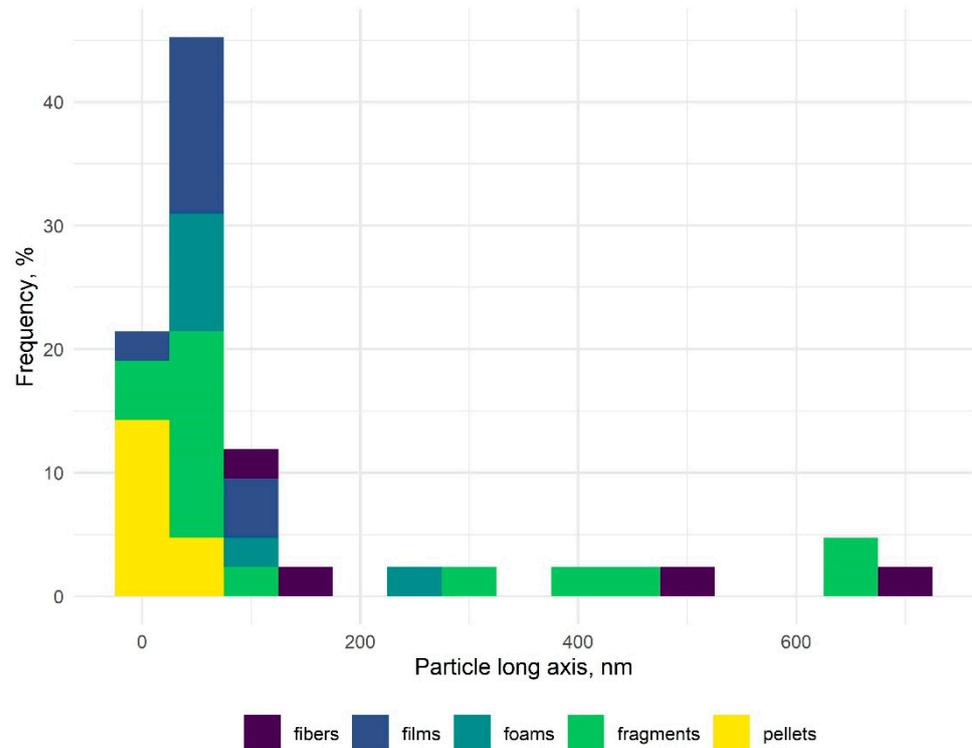


Figure 5. Particle size characteristics of microplastics. Each bar shows the contribution by microplastic type for each size class.

3.3. Microplastic Screening by SEM/EDS

SEM coupled with an energy dispersive X-ray unit was used to explore the surface morphology of microplastics. SEM images revealed different surface roughness in different microplastics, showing that the microplastics have complex surface topography characteristics generally characterized as rough, porous, cracked, or badly damaged. Fragmented microplastics were observed with various morphological diversity. Surfaces were rough or uneven, with obvious wear marks at the ends. Film microplastics comprised irregular films with light and soft textures. Most fibers displayed a smooth surface and linear shape. The foam microplastics had rounded shapes. Our results showed that microplastics have complex surface topography characteristics, generally characterized by rough, porous, cracked, and badly damaged surfaces due to the weathering degradation of plastics (Figure 6).

The EDS analysis indicated that Cl peaks characterized the microplastics identified in TL (Figure 7a) and DK (Figure 7b), i.e., these particles can be classified as polyvinyl chlorides [56]. In addition, Pb and Zn were present in 30% of the microplastic particles (Figure 7c), and Ti (Figure 7b) was found in 5% of the samples. The presence of Pb and Zn is associated with the possibility of the accumulation of these metals on microplastic particles, which has been previously proven both in a laboratory experiment and in natural aquatic systems [57]. Ti was present since it is usually added to plastics as a pigment or UV stabilizer [58]. Thus, both the greatest number of particles and the widest variety of metals in microplastics themselves have been identified in DK.

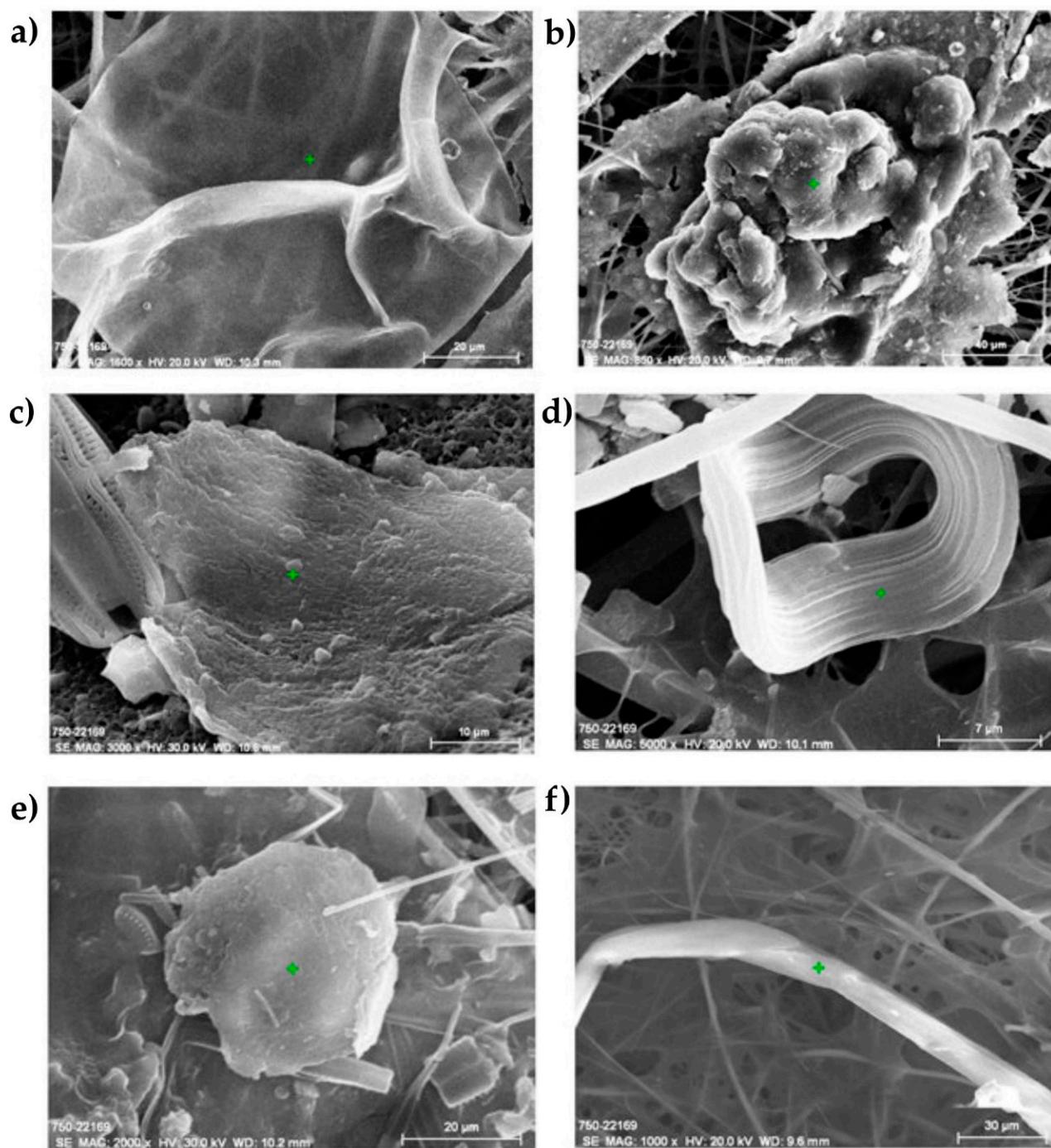


Figure 6. SEM images of the microplastic in Talmen Lake (a), Dzhulukul Lake (b), Teletskoye Lake (c), Zludyri Lake (d), Degtyarka Lake (e), Kuchuk Lake (f).

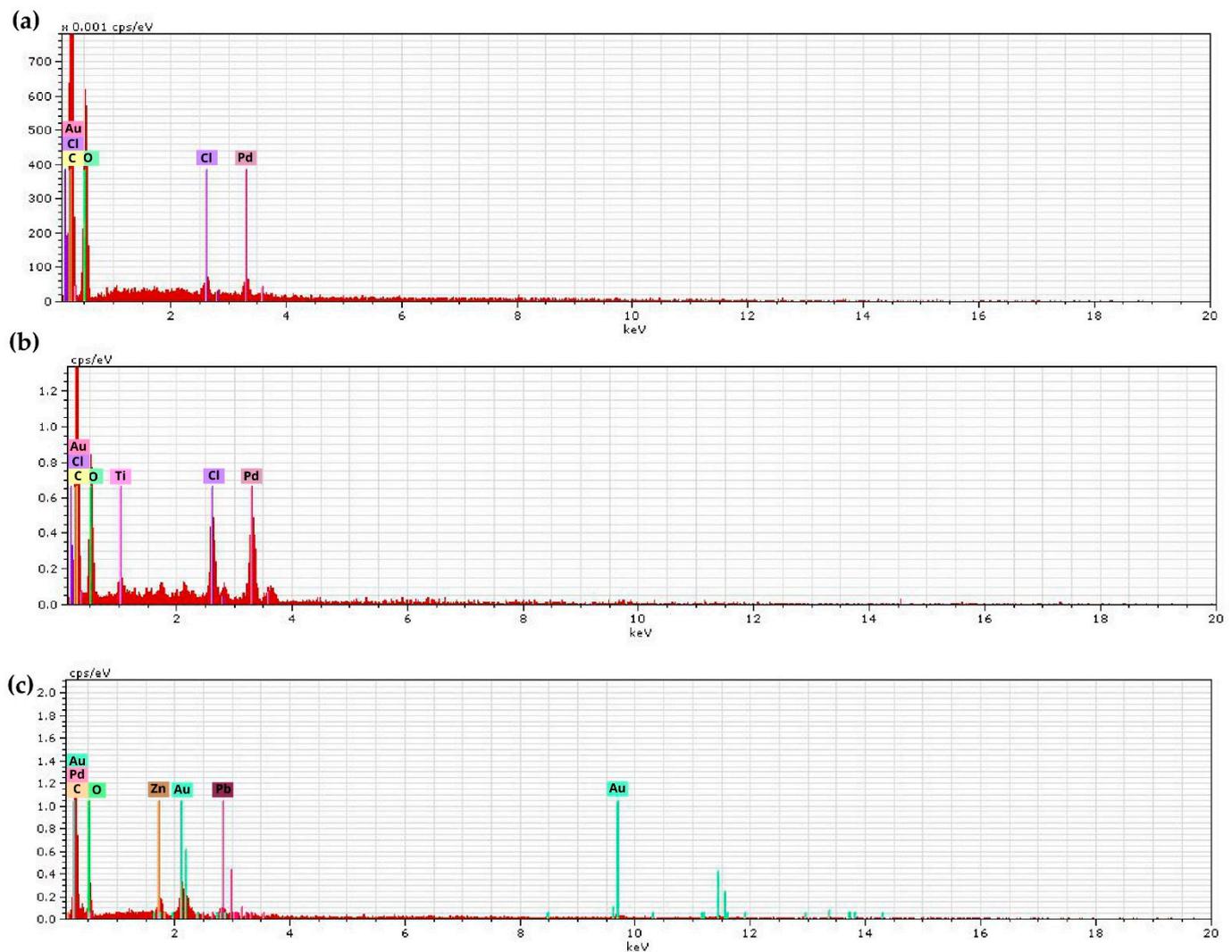


Figure 7. EDS spectrums of the microplastic in Teletskoye Lake (a), Zludyri Lake (b), Degtyarka Lake (c).

4. Conclusions

This study is the first reporting on microplastics in the South Siberian inland lakes. All the lakes contained MPs in water samples, with an average concentration value of 11 ± 7 MPs L^{-1} . Comparing with other inland lakes, South Siberian lakes presented moderate MPs concentrations. The minimum value was detected at remote ZL and shallow DZ (4 and 5 MPs L^{-1}) and the maximum at DK (26 MPs L^{-1}), a small endorheic lake with various human activities. Among the registered MPs forms, fragments and films were dominant, with a size range between 31 and 60 nm. The number of particles larger than 300 nm did not exceed 15%. The EDX analysis indicated Pb and Zn in 30% of the microplastic particles and Ti in 5% of the samples. These elements can harm both the environment and species. Microplastics have been identified even in remote lakes without permanent populations located within strictly protected areas. To a large extent, the concentrations and configuration of microplastics depend on local human activities (fishing, transport, landfilling). However, patterns associated with the proximity of the lake's inflow and outflow were also found. In TL, the concentration increased near the main tributary and decreased closer to the outflow, despite the increase in population on the shoreline. Overall, the present findings set a baseline and emphasize the need for increased attention to waste management and sustainable water use in Siberian freshwater environments.

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