Analysis of microplastic concentrations in water and bottom sediments as a new aspect of ecological monitoring

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Abstract. In this article we consider some features of spatial distribution of microplastic particles in water column and bottom sediments of Lake Ladoga based on results of research conducted in 2018-2019. This new type of contamination poses new threats to ecological conditions of water bodies. Considering the growing relevance of this problem, it is important to estimate genesis and scale of this contamination and its spatial and temporal distribution for developing recommendations for managing conditions of water bodies. It is advisable to add microplastic research including analysing microplastic concentrations and chemical composition of the particles to the system of ecological monitoring.

1. Introduction
There was a significant increase in plastic production since the last century. Different types of plastics have a wide range of characteristics that enable to replace some natural materials with them. Plastics in the broad sense are artificially synthesized high molecular compounds (polymers). These are different types of plastics, rubber, synthetic fabric. The most common types of plastics are polyethylene, polypropylene, polyvinyl chloride, polyethylene terephthalate [1].

Plastics are durable, light and their production costs are low. Thus, plastic production has been increasing year after year, and nowadays it has reached hundreds millions of tons per year. Considering the fact that decomposition period of plastics under natural conditions can be up to hundreds of years, the contamination of the environment by plastic particles is significantly growing [2, 3].

In aquatic environment microplastic particles become gradually covering by biofilms. These biofilms consist of microorganisms and humus matter and sorb toxic substances. Fish and other aquatic organisms misread these particles as food and ingest them. Thus, microplastics start to transfer in the food chain, finally comes to humans [4, 5]. Moreover, plastics themselves can contain some toxic components, such as phthalates and BPA [6], that have a potential negative impact on living organisms. A lot of new experiments confirm this negative impact of microplastics on organisms. For instance, the exposure of the blue mussel Mytilus edulis L. to microplastics resulted in histological changes and a strong inflammatory response [7], inflammation, oxidative stress and significant alterations in the gut microbiome were observed in the zebrafish [8], ingestion of microplastics by the monogonont rotifer (Brachionus koreanus) led to significant size-dependent effects, such as reduced growth rate, reduced fecundity, decreased lifespan and longer reproduction time [9]. At the same time, however, there is evidence that microplastic particles are not very harmful for organisms upon short-
term exposure [10]. Undoubtedly, such studies should be statistically proven, therefore, there is a need for additional research in this sphere.

The main risk of plastic contamination for ecosystems is that plastics are gradually breaking up to micro- and nano- sized particles, and it is almost impossible to remove them from the environment. Microplastic particles can be transported by wind and streams over long distances and nowadays scientists find them in most ecosystems all over the world [11, 12]. Institute of Limnology of the Russian Academy of Sciences started researching microplastic contamination of water bodies in Saint Petersburg, Leningrad Oblast and the Republic of Karelia in 2018. Special attention was given to Lake Ladoga and its catchment area.

Lake Ladoga is a largest lake in Europe and one of the most northern lakes among the greatest lakes of the world. The condition of the lake’s ecosystem is a result of complex interactions of processes in the lake and its catchment area under the influence of natural and anthropogenic factors [13]. Ladoga is a strategic object for Saint Petersburg as the lake’s water goes to water intakes of the city via the Neva River.

Institute of Limnology conducts studies of microplastic abundance in water and bottom sediments of Lake Ladoga and its catchment area. Concentration of plastic particles in water column is a result of different factors including hydrodynamic characteristics of the sampling site, seasons and weather. Bottom sediments act as deposit environment and therefore provide more adequate information about the long-term picture of the microplastic contamination of water body.

In this article we consider some features of spatial distribution of microplastic particles in water column and bottom sediments of Lake Ladoga based on results of research conducted in 2018-2019.

2. Materials and methods

Nowadays there is no unified method of sampling and processing water and bottom sediments for analysis of microplastic abundance.

Some groups of scientists use manta trawls to sample water and evaluate microplastic concentration in particles/km² [14]. Another method is pumping water through various filter devices and analysis of the particles settled on the filters. In this case concentration of microplastics is evaluated in particles/liter. There are also other units of measurement, for instance, gram/liter.

Bottom sediments analysis requires special sample processing, which can be realized in various ways. Different microscopes and various methods of spectrometry (Infrared or Raman spectrometry) are used during sample processing and analysis, that undoubtedly influences the results.

Thus, it is difficult enough to compare data on microplastic concentrations for different water bodies. The concentrations can be reliably compared if different studies are conducted using the same methods and preferably by one group of scientists.

In 2018 Institute of Limnology of the Russian Academy of Sciences conducted the first research of microplastics in the Gulf of Finland and several rivers of Saint Petersburg. Special attention was given to adaptation of existing methods of sampling and analysis to the local conditions [15].

Since 2019 regular measurements of microplastic concentrations in water column and bottom sediments have been made in Lake Ladoga and its catchment area by the same research group.

Within this framework water samples were taken using a special filter device (figure 1).
Water was filtered through a metal mesh with a mesh size of 60 μm. The number of filtered litres was recorded using an electronic counter. The construction of this device provides minimum contact of a sample with plastic details. All the particles settle on a metal mesh before pumping through a system of rubber pump hoses, that significantly reduces the risk of external contamination of filters.

Bottom sediments were sampled at different depths using the Ekman grab. In winter samples were taken from ice, in summer – from a board of research vessel (figure 2).

The analysis of microplastic concentration in bottom sediments requires a special sample processing. The process is based on the method developed in Shirshov Institute of Oceanology of the Russian Academy of Sciences [16]. This method was adapted for freshwater bodies.

The first stage was density separation with ZnCl₂ solution for separating mineral and organic matter from plastic particles. 400 g of dry sediment were placed into a glass beaker and 55% solution of ZnCl₂ was added (1.7 g/cm³), the sediment was stirred for 10 min. Then the sediment was left to settle for 1 hour (for sand) and 24 hours (for silt). After settling and deposition of solid mineral fraction, the supernatant with microplastic particles and organic matter was filtered through a metal...
mesh with a mesh size of 60 μm. Then total solids from the filter were rinsed with distilled water into a glass beaker for further wet peroxidation and removing of natural organic matter (fragments of living organisms, humus matter) [15].

To remove biological matter excluding impact on microplastic particles was used the method recommended by NOOA [17]. 30% hydrogen peroxide and Fe(II) catalyst solution were added into the glass beaker containing total solids filtered after settling. Then the beaker was placed into a water bath and heated up to 75 °C until organic matter was dissolved. After dissolving organic matter, the solution was filtered through a metal mesh with a mesh size of 60 μm into a glass beaker.

Centrifugation of the sample in NaCl solution was used for additional separation of microplastics. Particles settled on a mesh after filtration were rinsed with 26% NaCl solution into test tubes and then centrifuged for several minutes at the speed of 3000 rpm. Then the supernatant was filtered again through a metal mesh with a mesh size of 60 μm. The mesh was rinsed with distilled water to wash out the separation solution and then the total solids were rinsed with distilled water into a Petri dish.

Blank samples are used during sampling and processing to reduce false positives in the results. There are different methods to check external contamination: during sample processing some scientists place damp filter paper in Petri dishes and leave it exposed to airborne contamination [18], sometimes they use filter nets [19] or purified water [20]. Then blank samples can be treated in the same manner as real samples. The particles in the blank samples are subtracted from the total numbers of microplastic particles.

In this study a blank sample (distilled water) was used during all the stages of sample processing parallel to a real sample and in the same manner. The analysis of this blank sample did not show any external contamination.

Particles in a Petri dish were analyzed quantitively and qualitatively. Firstly, microplastic particles were identified using the light microscope. Recommendations [17, 21] were used for visual identification of microplastic particles.

During the analysis of samples under the light microscope it is particularly difficult to identify plastic fibers that can be similar to biological matter, for example, algae and chitin particles. The main distinguishing features of synthetic polymers are absence of cellular or organic structures, equal thickness throughout their entire length, heterogeneity of color. Plastic fragments often have irregular edges, however, biofilms on their surface and their partial destruction can make visual identification of these particles much more difficult. Therefore, the results should be additionally verified. It is strongly recommended to additionally analyze samples using methods of fluorescence microscopy [22] or spectrometry methods.

In this study Raman spectrometry was used to verify the results and identify chemical composition of the particles.

3. Results and discussion
The results of the analysis of microplastic concentrations in Lake Ladoga by Institute of Limnology in 2018-2019 are presented in figure 3 and figure 4.

Ladoga water is characterized by relatively low levels of different contamination, especially in northern parts of the lake where the depths are great. It relates also to microplastic concentrations both in water column and bottom sediments of the lake. The concentration of the particles within the size range in water column varies from 0.03-0.04 particles/liter in the western and central parts of the lake to 0.6 particles/liter near Pitkyaranta town where there are exceedances for many parameters of contamination. The concentration of microplastics in bottom sediments of the lake varies from 8-9 particles/kg in the western parts to 90 particles/kg in the area of Pitkyaranta town.

The results of microplastic concentrations are mapped on bathymetric map of Lake Ladoga [13]. No direct dependency was fixed for the concentrations results with the depth of sampling site of bottom sediments. However it was fixed a correlation between microplastic content and a point source of pollution both in water and bottom sediments, maximum concentrations of microplastics were found in the area near a functioning pulp mill in Pitkyaranta.
Figure 3. Concentrations of microplastic particles (particles/liter) in water column of Lake Ladoga.

Figure 4. Concentrations of microplastic particles (particles/kg) in bottom sediments of Lake Ladoga.
Some correlations between microplastic concentrations in water and bottom sediments of the lake can be preliminary fixed. However, this statement requires further research and analysis of the large data set.

The microplastic concentrations for Ladoga Lake were compared with the results of the study in the Gulf of Finland conducted by the same group of scientists. It shows that the Gulf of Finland is more contaminated by plastic particles than Ladoga. Microplastic concentrations in bottom sediments of the Gulf of Finland vary from 45 to 187 particles/kg [15], and these numbers are several times higher than were fixed in Lake Ladoga.

![Figure 5. Microplastic fiber from the sample of bottom sediments of Lake Ladoga.](image)

The analysis of the samples by Raman spectrometry was made to verify the results and identify chemical composition. Some particles were additionally analyzed under the electron microscope (figure 5). It was confirmed that there were different types of plastics in the samples of water and bottom sediments: the particles of polyethylene, polycarbonate and nylon were found. A large number of cotton fibers were also detected.

![Figure 6. The spectrum of the polyethylene terephthalate particle from the sample of bottom sediments of Lake Ladoga.](image)
The prevalent type of microplastics in Ladoga samples was polyethylene terephthalate (figure 6). It can possibly be particles of degraded plastic bottles and synthetic fibers releasing during the washing of clothes.

Analysis of chemical composition of microplastic particles is important as verification method and also because it helps to make some conclusions about sources of plastic contamination, genesis of the particles, its transportation and transformation in water objects.

4. Conclusion
According to the results of recent studies, water column and bottom sediments of water object are permanently contaminated by microplastics particles. This new type of contamination poses new threats to ecological conditions of water bodies. Different aspects of this environmental problem require further detailed research. Considering the growing relevance of this problem, it is important to estimate genesis and scale of this contamination and its spatial and temporal distribution for developing recommendations for managing conditions of water bodies. It is advisable to add microplastic research including analyzing microplastic concentrations and chemical composition of the particles to the system of ecological monitoring. To fully understand scales and dynamics this type of contamination, there is also a need for a large data set that can be obtained only with regular studies.

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References
[1] Geyer R, Jambeck J R and Law K L 2017 Production, use, and fate of all plastics ever made Science Advances 3(7) 5 p
[2] Andrady A L 2011 Microplastics in the marine environment Marine Pollution Bulletin 62(8) pp 1596–1605
[3] Thiel M and Gutow L 2005 The ecology of rafting in the marine environment. II. The rafting organisms and community Oceanography and Marine Biology 43 pp 279–418
[4] UNEP 2016 Marine Plastic Debris and Microplastics — Global Lessons and Research to Inspire Action and Guide Policy Change Nairobi: United Nations Environment Programme 252 p
[5] GESAMP 2016 Sources, Fate and Effects of Microplastics in the Marine Environment: Part Two of a Global Assessment 93 220 p
[6] Hansen E, Nilsson N H, Lithner D and Lassen C 2013 Hazardous Substances in Plastic Materials COWI and Danish Technological Institute 149 p
[7] Von Moos N, Burkhardt-Holm P and Koehler A 2012 Uptake and effects of microplastics on cells and tissue of the blue mussel Mytilus edulis L. after an experimental exposure Environmental Science & Technology 46 pp 327–35
[8] Qiao R, Sheng C, Lu Y, Zhang Y, Ren H and Lemos B 2019 Microplastics induce intestinal inflammation, oxidative stress, and disorders of metabolome and microbiome in zebrafish Science of The Total Environment 662 pp 246-53
[9] Jeong C B, Won E J, Kang H M, Lee M C, Hwang D S, Hwang U K, Zhou B, Souissi S, Lee S J and Lee J S 2016 Microplastic size-dependent toxicity, oxidative stress induction, and p-JNK and p-p38 activation in the monogonont rotifer (Brachionus koreanus) Environmental
[10] Selonen S, Dolar A, Kokalj A J, Skalar T, Dolcet L P, Hurley R and Van Gestel C A 2020 Exploring the impacts of plastics in soil—The effects of polyester textile fibers on soil invertebrates *Science of The Total Environment* **700** P 134451

[11] Corcoran P L, Biesinger M C and Griffi M 2009 Plastics and beaches: A degrading relationship *Marine Pollution Bulletin* **58(1)** pp 80–4

[12] Barnes D K, Galgani F, Thompson R C and Barlaz M 2009 Accumulation and fragmentation of plastic debris in global environments *Philosophical transactions of the Royal Society of London Series B, Biological sciences* **364(1526)** pp 1985–98

[13] Rumyantsev V A 2015 *Ladozhskoe ozero i dostoprimechatel'nosti ego poberezh'ya. Atlas [The Atlas ‘Lake Ladoga and the coastal remarkable sights’]* Saint Petersburg: Nestor-Historia 200 p

[14] Il'ina O V, Kolobov M Y and Il'inskii V V 2021 Plastic pollution of the coastal surface water in the middle and southern Baikal *Water Resources* **48(1)** pp 56-64

[15] Pozdnyakov Sh R, Ivanova E V, Guzeva A V, Shalunova E P, Martinson K D and Tikhonova D A 2020 Studying the concentration of microplastic particles in water, bottom sediments and subsoils in the coastal area of the Neva Bay, the Gulf of Finland *Water Resources* **47(4)** pp 599–607

[16] Zobkov M and Esiukova E 2017 Microplastics in Baltic bottom sediments: Quantification procedures and first results *Marine Pollution Bulletin* **114** pp 724–32

[17] Masura J, Baker J, Foster G and Arthur C 2015 *Laboratory Methods for the Analysis of Microplastics in the Marine Environment: Recommendations for quantifying synthetic particles in waters and sediments* NOAA Technical Memorandum NOS-OR&R-48 P 31

[18] Cunningham E M, Ehlers S M, Dick J T A, Sigwart J D, Linse K, Dick J J and Kiriakoulakis K 2020 High abundances of microplastic pollution in deep-sea sediments: evidence from Antarctica and the Southern Ocean *Environmental Science & Technology* **54(21)** pp 13661–71

[19] Zobkov M, Belkina N, Kovalevski V, Zobkova M, Efremova T and Galakhina N Microplastic abundance and accumulation behavior in Lake Onego sediments: a journey from the river mouth to pelagic waters of the large boreal lake *Journal of Environmental Chemical Engineering* **8(5)** P 104367

[20] Tamminga M and Fischer E K 2020 Microplastics in a deep, dimictic lake of the North German Plain with special regard to vertical distribution patterns *Environmental Pollution* **267** P 115507

[21] Marine & Environmental Research Institute 2015 *Guide to Microplastic Identification* P 13

[22] Ivanova E V, Guzeva A V, Lapenkov A E, Pozdnyakov Sh R, Kapustina I L, Mitrukova G G and Tikhonova D A 2020 Osobennosti primeneniya krasitelya Nil'skij krasnyj dlya identifikacii chastic plastika v prirodnym ob"ektah [The aspects of using Nile Red for the detection of plastic particles in the environment] *Russian Journal Of Applied Ecology* **4(24)** pp 36–42