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What to monitor? Microplastics in a freshwater lake – From seasonal surface water to bottom sediments

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ABSTRACT

Marine microplastics have received considerable attention, and efforts are underway to develop standardised methods for sampling, sample treatment, and analysis, while the observation of freshwater ecosystems remains relatively overlooked. To address this understudied environment, we present a comprehensive case study on microplastics in an urban lake from Baltic region of Northern Europe covering the seasonal dynamics of microplastics in surface water, deposition rate throughout one year in sediment traps and distribution of microplastics in dated sediment archive to determine the most representative environmental compartment for microplastic pollution monitoring. The following well-established microplastic research methods have been used: Manta trawling for surface water, trapping for assessing microplastics sedimentation rate and coring for sediments. Attenuated total reflection and micro-Fourier transform infrared spectroscopy methods were used to investigate the synthetic nature of identified particles. The sediment core chronology was based on ^{210}Pb and Bayesian Plum model revealing sediment layers to represent even the time before the beginning of plastic mass production (approximately 1950). The surface water microplastic concentrations were higher in summer (5.71 particles/m^3) and gradually decreased towards winter (0.75 particles/m^3); they were almost 25 times higher in more recent (2018) sediments than in the deeper layers referring to years prior to 1890. Surprisingly, microplastic particles were found in sediments before the year 1950. The microplastic deposition rate was 9.47 particles/cm^2/year or 4.31 μg/cm^2/year. The most abundant polymers were polyethylene, polystyrene and polypropylene, and the prominent particle shapes were fibres in surface water and fragments in sediments. Our results provide a baseline for evaluating future contamination level changes in highly urbanized area. We recommend the combination of surface water filtering with net and sediment trapping methods for monitoring microplastics in lakes since this method requires little time and financial resources for sampling and processing and produces information on temporal microplastic occurrence and deposition.

1. Introduction

Microplastics (MPs) are considered ubiquitous and persistent pollutants found even in the most remote and pristine parts of the world due to being easily transported long distances (Allen et al., 2021; Aves et al., 2022; Bergmann et al., 2019). Once originated from the terrestrial environment, they can be carried by wind, rain and surface runoff, eventually reaching waterbodies (Horton and Dixon, 2018; Zalasiewicz et al., 2016). Rivers act as major pathways for MPs to lakes and oceans, where some of the MPs remain floating on water surface or suspended in

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the water column, while others are subjected to vertical transport and burial in sediments (Atugoda et al., 2020; Dai et al., 2018; Forrest et al., 2020; GESAMP, 2019; Horton and Dixon, 2018; Li et al., 2023a; Zalasiewicz et al., 2016). Bioturbation or particle morphology can further promote greater MP transport in sediments (Dimante-Deimantovica et al., 2024; Gebhardt and Forster, 2018). Moreover, the occurrence, dynamics and accumulation of MPs in particular environmental matrices depend not only on water ecosystem descriptors (hydrological, meteorological, topographical and morphometric) but also on MP physicochemical characteristics (size, shape, density, additives) that can cause particles to be unevenly distributed in the aquatic environment (Atugoda et al., 2020; Forrest et al., 2020; Kumar et al., 2021; Shamskhany et al., 2021). MPs contain a variety of chemical additives that may threaten the health and wellbeing of individual organisms and food chains (Bhuyan, 2022; Wagner et al., 2014). Therefore, assessing the presence and amount of MPs reflecting changes over time (i.e. monitoring of pollution level) is highly important.

Throughout the last decade marine environment has been studied extensively for microplastics. Although lately freshwater contamination with MPs has received increasing attention, studies focusing on lake and estuary ecosystems are still relatively scarce (D'Avignon et al., 2021; Lu et al., 2021; Nava et al., 2023). Simultaneously, the reported MPs content in freshwater and estuarine organisms are comparable to or even higher than marine organisms (Covernton et al., 2021; D'Avignon et al., 2021). Apart from biota and biological characteristics, lakes' ecosystems are impacted by different physio-chemical and geological processes that play a role in MPs circulation and distribution. The detailed analysis of MP contamination in different environmental compartments across seasons characterised by varying hydrological conditions can help to understand the potential risks to aquatic organisms and human health, as well as is crucial for developing mitigation strategies to protect both environmental and public health and setting threshold values. Hence, regular monitoring of MPs in lakes would allow to improve European (and beyond) freshwater quality providing comparison of waterbodies across Europe and facilitating decision making on quality status as to MPs pollution.

There is an urgent need to understand the spatiotemporal pattern of MP contamination since policyholders request reliable guidelines for the implementation of MP monitoring in different environmental settings (Backhaus, 2023). The latest proposals by the EU Commission for Amending Water Legislation Directive (i.e., Water Framework Directive (2000/60/EC), Groundwater Directive (2006/118/EC) and Environmental Quality Standards Directive (2008/105/EC)) clearly state that MPs are potentially harmful and should be included in surface and groundwater watch lists and monitored to set environmental quality standards (EQS) as soon as suitable monitoring methods are identified (Backhaus, 2023). Moreover, the identification and development of guidelines and methodologies, as well as the addition of MPs to the existing EQS list, are planned for the near future (Backhaus, 2023; Directorate-General for Environment, 2022). Therefore, the most time-efficient, cost-efficient and reliable data-efficient methods for assessing MP pollution in freshwater ecosystems are of urgent importance and a subject of discussion (UNEP, 2020; Zhang et al., 2022). As suggested by D'Avignon et al. (2021), MPs concentration should be considered as an ecologically relevant parameter – included in standard sampling protocols and water quality assessments. Nevertheless, the origin, accumulation rate and temporal changes of MPs in lakes are poorly understood, hampering research-based decision-making for pollution mitigation (Chen et al., 2024; D'Avignon et al., 2021).

Lake surface water and sediments are among most common environmental compartments attracting the interest of researchers (Pan et al., 2023). For surface water sampling several devices have been used, however, some established sampling methods, such as Manta net trawling, are among most common for particles in sizes 300 µm and above. According to Pasquier et al., (2022) the mesh size of 300-350 µm is the most used in both marine and freshwaters, which is an optimal compromise between representativeness of the sample and possible net clogging due to plankton organisms (Nayebi et al., 2023; Stock et al., 2019). Based on research goal, pump systems with finer mesh might be used to acquire information on smaller-sized particles for more accurate assessment of environmental impact (Chen et al., 2024). Since only part of MPs is floating in the surface waters and the buoyancy might lessen over time initiating particle downward transport, it is crucial to study MPs presence and accumulation in sediments. Sediments are considered to be an important long-term sink of MPs, and the abundance of buried MPs varies greatly based on location in relation to pollution sources and the surrounding area of the waterbody (Chen et al., 2024; Enders et al., 2019; Malla-Pradhan et al., 2022; Warrier et al., 2022). As to sediments, several devices – corers and grab samplers – can be used to collect samples from different areas and depths, most commonly upper 5 cm of sediments are used for MP analyses. Some studies also collect vertical sediment cores to assess the MPs distribution throughout sediment layers (Nayebi et al., 2023). However, most studies have reported MPs in bulk sediment samples without assessing the age of the sediments, while only a few have reflected the MP accumulation history in dated subsurface lacustrine sediment cores (Dimante-Deimantovica et al., 2024; Dong et al., 2020; Huang et al., 2022; Li et al., 2023b). There are certain pitfalls using grab samples and cores that may lead to potential artefacts, i.e. replication (correlation among several parallel samples) might be difficult, sediments can be mixed and large variation in accuracy and precision of dating results can occur depending on the dating methods and the characteristics of sediments. Furthermore, dating is also time and cost demanding (Nayebi et al., 2023) or not possible at all (e.g. grab samples). This complicates the spatial and temporal interpretation of MPs' concentrations as well as comparison between different locations. Recently, Saarni et al., (2021, 2023) has applied a novel approach in MPs research by applying the well-established sediment trapping method for assessing MPs accumulation rate and understanding seasonal trends in MP accumulation fluxes, revealing that changes in MP accumulation rates are related to seasonal conditions. The advantage of sediment traps is the possibility to assess MPs vertical flux rate (i.e. number MP particles or MP weight per area per time unit) that gives direct information on the accumulation of MPs with high temporal resolution (seasonal variation), excluding the changes caused by varying overall sedimentation rate. The challenges in sediment trapping typically relate to small sample volumes, and the required resources for monitoring campaigns of long time periods.

In our study we have set a baseline for MP pollution temporal data and looked for most feasible monitoring methods comparing three approaches described above (surface water sampling, sediment core and sediment traps) in order not to compromise data quality. The objectives of this study were to (1) analyse the abundance, size, shape and colour of MP contamination in lake surface water, dated sediment core and sediment traps, (2) evaluate MPs seasonal occurrence in surface water and deposition rate in sediments, and (3) provide suggestions for feasible (quick, cheap and accurate) MP monitoring methods in lake ecosystems by choosing the most appropriate abiotic environmental compartment representing MP pollution during certain time periods and trends in the long term. Our data present evidence on MP pollution in a lake located in a highly urbanised and historically industrialised area. To the best of our knowledge, no previous studies have comprehensively investigated MP pollution in lake surface water and sediments while correlating it to the deposition rate over certain time period.

2. Materials and methods

2.1. Study area

The study was carried out in Lake Velnezers, which is located in Riga, the capital city of Latvia (Fig. 1). It is a eutrophic lake with a surface area of 3.5 ha, and its maximum and average depths are 7.4 m and 4.0 m, respectively (Dručka, 2014). It has no inflow or outflow streams and

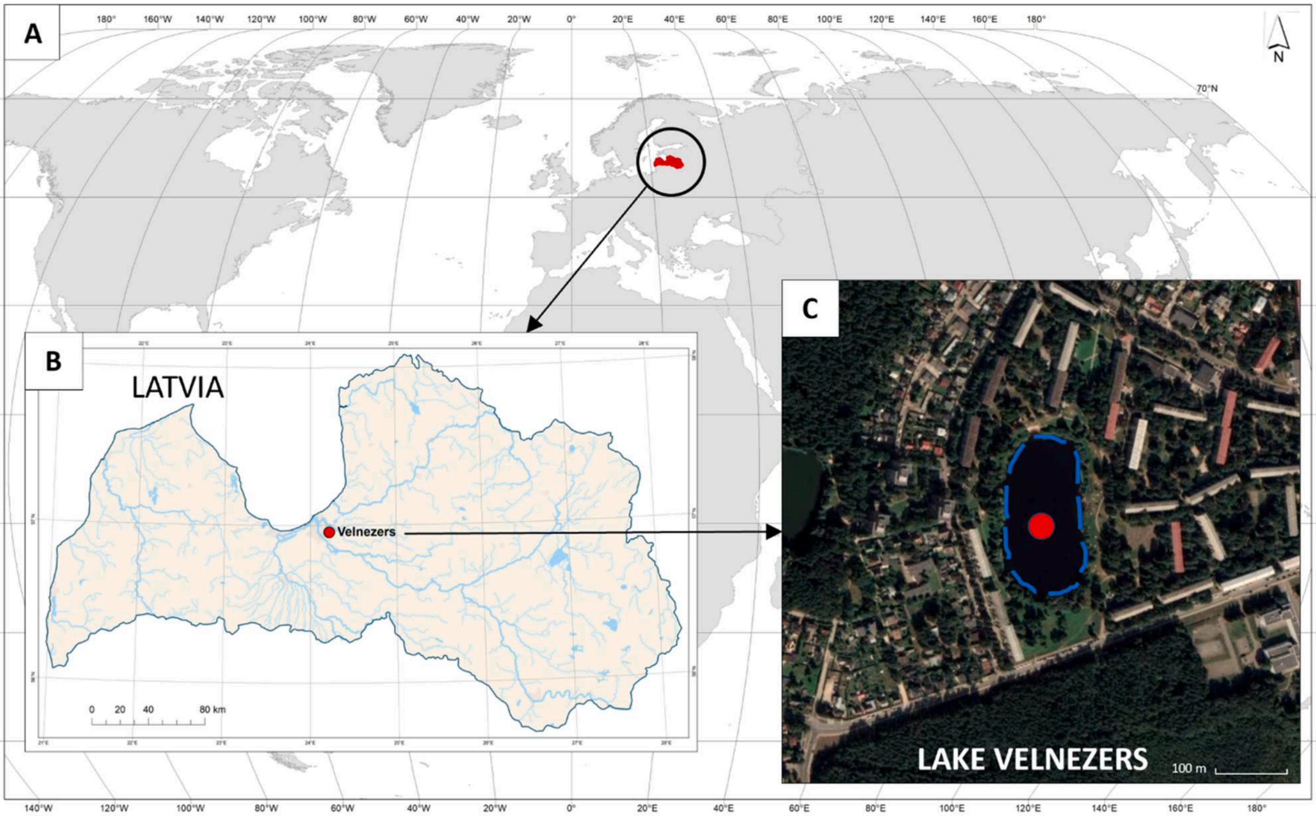

Fig. 1. Study area location within northern Europe (A), central Latvia (B), and Lake Velnezers (C), with the red dot marking the deepest part of the lake and dashed blue line the shore of lake.

feeds mostly on groundwater (Lūmane, 1998; Pujāte, 2015). Over the course of the 19th century, industry developed rapidly in the area adjacent to Lake Velnezers – several factories were built that produced, e.g., sugar, linen, paper, matches, barrels, leather and chemicals (Cita Rīga, 2020). Most factories were closed at the beginning of World War II, and only a few resumed their manufacturing – the area was especially marked as a textile industry district. During the period of industrialisation, most of the surrounding inland dune pine forest was cleared (Dodies.lv, 2022a, 2022b). The construction of private housing started in the 1950s, and intensive building of residential block houses was initiated in the 1960s. A few years later, the density of buildings reached the existing level, with private and residential block houses as close as 30 m and major city roads at a 75 m distance (Lūmane, 1998). Historically, Lake Velnezers has been used as a wastewater discharge site; consequently, there are increased concentrations of dissolved inorganic nitrogen and phosphorus substances (Dručka, 2014) and zinc (Lanka et al., 2024), causing eutrophication of the lake. The population in the Lake Velnezers neighbourhood is approximately 23,000, while more than 614,000 permanent residents inhibit the city itself (Centrālā statistikas pārvalde, n.d.). Currently, lake is used extensively by both locals and visitors as a recreational area.

2.2. Sampling

Seven surface water samples were collected once every six weeks from April 2019 to January 2020. It was performed by trawling a Manta net (Hydro-Bios, mesh size 300 μm, net material – nylon) from the stern part of the boat outside the wake zone for 20 minutes, resulting in a filtered water volume of 24.23-29.33 m^3 (on average 27.23 m^3), measured for each sample using mechanical flow metre (Hydro-Bios). After trawling, the net was rinsed from the outside to concentrate the sample in the cod end, which was then removed over a metal bowl, inverted and rinsed with distilled water. The sample was transferred to a glass tray, covered with a metal lid and kept at 2-4°C until processing.

The sediment trap consisted of two collector tubes, flotation buoys, a directing wing and an anchor, as described by Saarni et al., 2021. The collector tubes and tube bottom parts were made of polyvinyl chloride (PVC) plastic and stainless steel, respectively. The tube had an inner diameter of 5.6 cm. It was installed in February 2019 near the deepest part of the lake where sediment resuspension is minimal. The trap was left just above the bottom of the lake for six months and emptied, acquiring sample representing spring/summer (productive) season. Then the trap was repeatedly placed in the same place on August 2019, and left for five months. The second and final trap sample acquisition was performed in January 2020, acquiring sample representing autumn/winter (stagnant) season.

Sediment core samples were obtained in February 2019 in the deepest part of the lake (56.975949 N°, 24.247147 E°) using a Kayak/HTH gravity corer fitted with a polyvinyl chloride (PVC) sample tube with an 8.2 cm internal diameter. The total length of the core was 32 cm, which was further sliced into one cm layers to increase the resolution of sample representativeness and ease sample treatment and subsampled for further analyses. Afterwards, each 2 consecutive 1 cm sediment layers were fused and transferred to precleaned glass trays, covered with foil and metal lids and stored at 2-4°C until processing.

2.3. Chronology – sample preparation and analysis

The sediment core from Lake Velnezers was dated with ^{210}Pb at the University of Gdańsk following a standard procedure (Tylmann et al., 2016). Alpha spectrometry measurements of ^{210}Po were used to indirectly determine the activity of total ^{210}Pb. Sediment samples of 0.2 g were dried, homogenised and transferred into Teflon containers, spiked with a ^{209}Po yield tracer and digested with concentrated HNO_3, $HClO_4$

and HF at a temperature of 100°C using a CEM Mars 6 microwave digestion system. After 24 hours, the solution was transferred to a Teflon beaker, evaporated with 6 M HCl to dryness and dissolved in 0.5 M HCl. Within four hours, polonium isotopes were spontaneously deposited on silver discs, and the discs were analysed for ^{210}Po and ^{209}Po using a 7200-04 APEX Alpha Analyst integrated alpha-spectroscopy system (Canberra) equipped with PIPS A450-18AM detectors. The samples were counted for 24 hours. A certified mixed alpha source (^{234}U, ^{238}U, ^{239}Pu and ^{241}Am; SRS 73833-121, Analytics, Atlanta, USA) was used to check the detector counting efficiencies, which varied from 30.9% to 33.9% for the applied geometry. The ages of the sediment layers are presented as the means for each centimetre.

For the core Bayesian age-depth modelling *Plum* was built in an R environment via 'rplum' package (Aquino-López et al., 2020). *Plum* is a Bayesian forward model that integrates two different processes, in this case, the behaviour of the ^{210}Pb flux and the variation of ^{210}Pb with depth, and an age-depth function (Blaauw and Christen, 2011). Compared to the traditional implementation of the Constant Flux (or Constant Rate of Supply) model, the recently developed Bayesian *Plum* model is more flexible forward model allowing to cope with imperfect ^{210}Pb depth profiles (e.g., can easily handle gaps and include other types of dating information) providing an estimate of most likely age distributions. The precision of *Plum* Bayesian age-depth model can be increased through elevated dating densities; the model 'learns' to produce more accurate and precise chronology during the modelling process, whereas classical linear interpolation does not (Aquino-López et al., 2020).

2.4. Sediment characteristics – loss on ignition

The relative content of organic matter in the sediments was determined by loss on ignition at 550°C for four hours. The relative mineral matter and carbonate contents were estimated based on the weight loss after sample combustion at 550 and 950°C for two hours. Because the weight loss after 950°C is the amount of CO_2 evolved from carbonate minerals, to obtain the actual percentage of CO_3^{2}, the weight after 950°C combustion was multiplied by 1.36 (Dean, 1974; Heiri et al., 2001).

2.5. Sample purification for microplastic extraction

MPs were isolated from samples by applying several step treatment protocols (Table 1) using oxidation, surfactant extraction, enzymatic digestion (with cellulase, viscozyme in acetate buffer (pH 4.8) and alcalase, protease in TRIS buffer (pH 8.2)) and density separation (with sodium polytungstate solution, density 1.9 g/cm^3) (Suppl. Mat. Section 1).

2.6. Quality control and quality assurance

Throughout sample handling, precautionary measures were taken to avoid unintentional sample contamination or loss (Suppl. Mat. Section 2). Procedural negative control samples were treated and analysed in parallel with each sample series to assess possible sample contamination. Particle recovery (positive control) during treatment was tested in triplicate by spiking water matrix and sediment samples with 100 standardised red polystyrene (PS) beads (⌀ 100 μm, density 1.05 g/cm^3, Sigma–Aldrich, product no. 56969-10ML-F). The spiked samples were treated in the same manner as the field samples, except for the water matrix, where a 50 μm sieve instead of a 200 μm sieve was used.

2.7. Microplastic characterisation and polymer analysis

The surface water MP samples were filtered through glass fibre filters (pore size of 1.2 μm). The sorting and counting of potential plastic particles (≥300 μm) were carried out under a Leica DM400 B LED light microscope fitted with a DFC 295 camera and Leica Application Suite V4.1 software via visual identification. The particles were visually categorised according to their shape (fibres or fragments) and colour, and measured by their maximum and minimum dimensions. Particles of sizes suitable for manual handling were picked out using tweezers and further identified using Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR) spectroscopy (Thermo Scientific, Nicolet iS20) and OMNIC software, which contained 30 libraries with more than 15000 synthetic and non-synthetic polymer spectra. To test the synthetic nature of particles not suitable for spectroscopic analysis by ATR-FTIR, a "hot needle" test was applied (Postma, 2022); from every sample filter, up to the first 10 encountered particles were tested. The mass of surface water MPs was not determined.

Sediment material MPs <500 μm in size separated from the sediment trap and core samples via size fractionation were vacuum filtered through silver membrane filters (pore size 5 μm, ⌀ 12 mm, Sterlitech Co.). The filtered and analysed volume was 29-100 v% per sample, depending on the total solid content. The filtered volume was optimised to prevent stacking of particles, which may lead to underestimated numbers of MPs. The whole filtered area of the silver membranes was measured with an FTIR spectrometer (Agilent Cary 670/620) fitted with a focal plane array (FPA) detector in reflection mode as described previously by Uurasjärvi et al., 2021. The measurement parameters were as follows: 15x cassegrain objective, 8 cm^{-1} spectral resolution, 4 scans, 3800–800 cm^{-1} spectral range, and 5.5 μm pixel resolution. The data were analysed with siMPle software (Primpke et al., 2020) and a spectral library of the most common plastic and non-synthetic polymers: polyethylene (PE), polypropylene (PP), polyamide (PA), polyethylene terephthalate (PET), polystyrene (PS), acrylonitrile butadiene styrene (ABS), polyurethane (PU), polyvinyl chloride (PVC), polymethyl methacrylate (PMMA), polyacrylonitrile (PAN), cotton and proteins. The library and settings for analysis in siMPle were as described by Uurasjärvi et al. (2021). SiMPle automatically counts the number of MPs, identifies their polymer, measures the Feret diameter and estimates the mass of the MPs.

To determine the particle shape (or elongation) on a continuous scale, the aspect ratio (AR) was calculated for each individual particle by the relation of the minor and major Feret diameters. Since the shape of particles ≥300 μm (water samples) was determined visually and that of smaller particles (<500 μm in the sediment trap and core samples) was determined numerically, the surface water particle third quartile AR value (0.11) was also applied to small particles. The shapes of the small particles were therefore categorised into fibres (AR ≤0.11) and

Table 1
Treatment steps applied for extracting microplastics from water, sediment trap and sediment core samples.

Samples	Treatment steps								
	Freeze-drying	30% H_2O_2	Density separation I	Size fractionation with 500 μm sieve	5% SDS	Enzymes in TRIS	Enzymes in acetate	Fenton	Density separation II
Water		X							
Sediment traps		X	X	X					
Sediment core	X	X	X	X	X	X	X	X	X

fragments (AR >0.11). To assess the connection between particle elongation and position in the sediment profile, the AR was further divided into four classes—AR<0.25, 0.25≤AR<0.5, 0.5≤AR<0.75, and 0.75≤AR≤1—to determine the proportional differences among the mentioned groups in each sediment layer.

2.8. Statistical analysis

The correlation between the AR of the particles (representing elongation) and their position within the sediment layers, as well as the relationship between the sediment bulk density and MP concentration within the corresponding sediment layers, was evaluated using R software (R Core Team, 2020). To assess the normality of the data distribution, the Shapiro−Wilk test was first conducted. Following the normality assessment, either Pearson's or Spearman's rank correlation test was employed, depending on the distribution of the data and the nature of the variables being analysed, with a 95% confidence level.

3. Results

3.1. Sediment Chronology

The profile of total ^{210}Pb concentration activity shows a surface mixing zone at depths ranging from 0-7 cm and a consistent decrease with sediment depth (Fig. 2A). The concentration activity measured in the lowermost sample was assumed to represent the supported ^{210}Pb level (45.5±3.4 Bq/kg). The excess ^{210}Pb profile displays a decrease downcore documented by an exponential function (R^2=0.93; Fig. 2B). This proves that there was no major disturbance to the sediment column. However, due to the surface mixing zone we decided to use Plum model which is more flexible and allows coping with non-ideal ^{210}Pb depth profiles (Aquino-López et al., 2020). The maximum age of 129 +24/-36 years (1890 +24/-36) was estimated at a sediment depth of 29 cm (Fig. 2C); dating of deeper layers was not possible due model restrictions. At a depth of 24 cm, the sediment age was calculated to be 64 +9/-6 years (1955 +9/-6).

3.2. Sediment characteristics

Lake Velnezers sediment core contains relatively low carbonate amounts throughout the studied time period, ranging from 1.76% (year 2019) to 4.21% (year 1998). Greater variability was observed for the organic and mineral matter contents, with shifts ranging from 40.40–55.15% and 43.1–57.03%, respectively (Suppl. Mat. Fig. S1A). There is a clear increasing trend in the organic matter content under the present conditions, with an organic matter content as high as 55.15%. The bulk density of the sediments gradually increased up to the layer from 2001-1998 (11-12 cm) and then remained relatively consistent throughout the core, varying from 0.085 to 0.245 g/cm^3 (on average 0.171 g/cm^3) (Suppl. Mat. Fig. S1B).

3.3. Microplastic positive and negative controls

Three water matrix procedural blank samples altogether contained 7 fibres; no fragments were identified. The particles were black (n=3), purple (n=2), blue (n=1) and pink (n=1) in colour, with sizes varying from 300 to 7450 μm. Surface water blank samples indicate up to 1.13% total background contamination, accounting for low unintentional sample contamination.

The sediment material procedural blank samples contained 72 fragments (on average 24.00 ±11.53 particles, with a maximum of 35 particles per blank sample); no fibres were identified. The size of the MPs varied from 51.7 to 595.8 μm. They were represented by the following polymers: polyethylene terephthalate (PET, 91.55%), polymethyl methacrylate (PMMA, 2.82%), polypropylene (PP), polyethylene (PE), and polyacrylonitrile (PAN) (in total constituting 5.63%). Because of high background contamination by PET and equipment containing the PMMA polymer being used occasionally during sample handling, particles consisting of the mentioned polymers were excluded from the dataset. A particle consisting of PAN was identified in the blank sample, but this polymer type was not found in the core or trap samples. There were no particles matching the polymers of the sampling equipment (PVC tubes for trap and core or nylon for Manta net) found in the samples. The background pollution by PP and PE was considered negligible since only two PP particles and one PE particle were found. The sediment material blank samples indicate up to 1.39% (trap samples) and 0.33% (sediment core samples) total background contamination.

The surface water and sediment trap treatment positive control samples yielded average recovery rates of 93.28 ±1.84% and 91.73 ±0.70%, respectively, while for the sediment core, the recovery rate was 78.95 ±3.95%.

The acquired results for environmental MPs were not corrected based on positive and negative control data due to relatively low background contamination, other than the exclusion of PET and PMMA polymer particles from sediment material samples.

3.4. Microplastics in surface water

MPs ≥300 μm were present in all collected surface water samples, with an average concentration of 3.30 particles/m^3. The concentrations were greater at the beginning of the summer (5.71 particles/m^3) and gradually decreased towards early winter (0.75 particles/m^3) (Fig. 3A). Fibres were the most common MP particle shape (95.65%), while

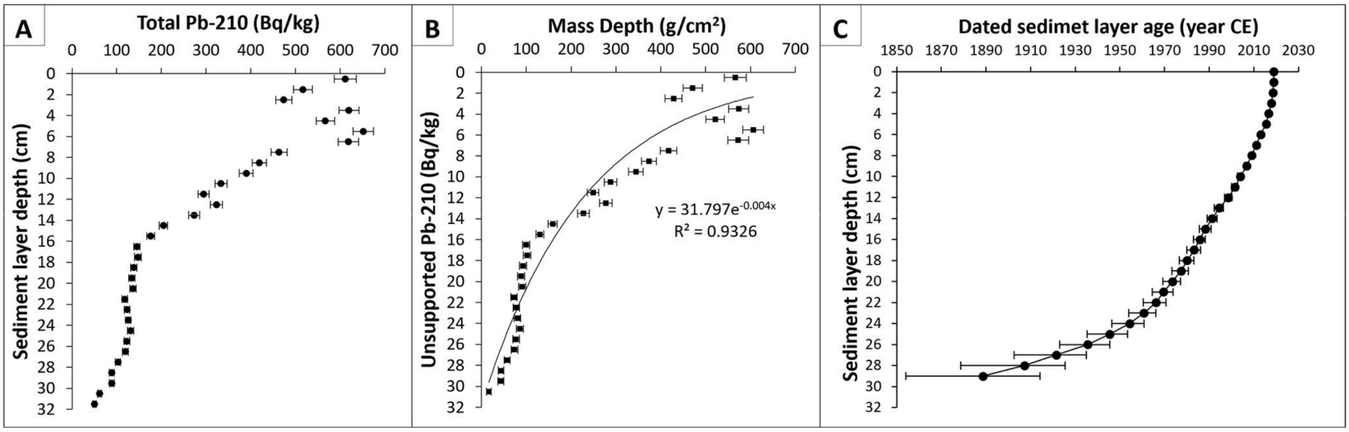

Fig. 2. Lake Velnezers (A) depth profile of total ^{210}Pb; (B) depth profile of excess ^{210}Pb; (C) age-depth model.

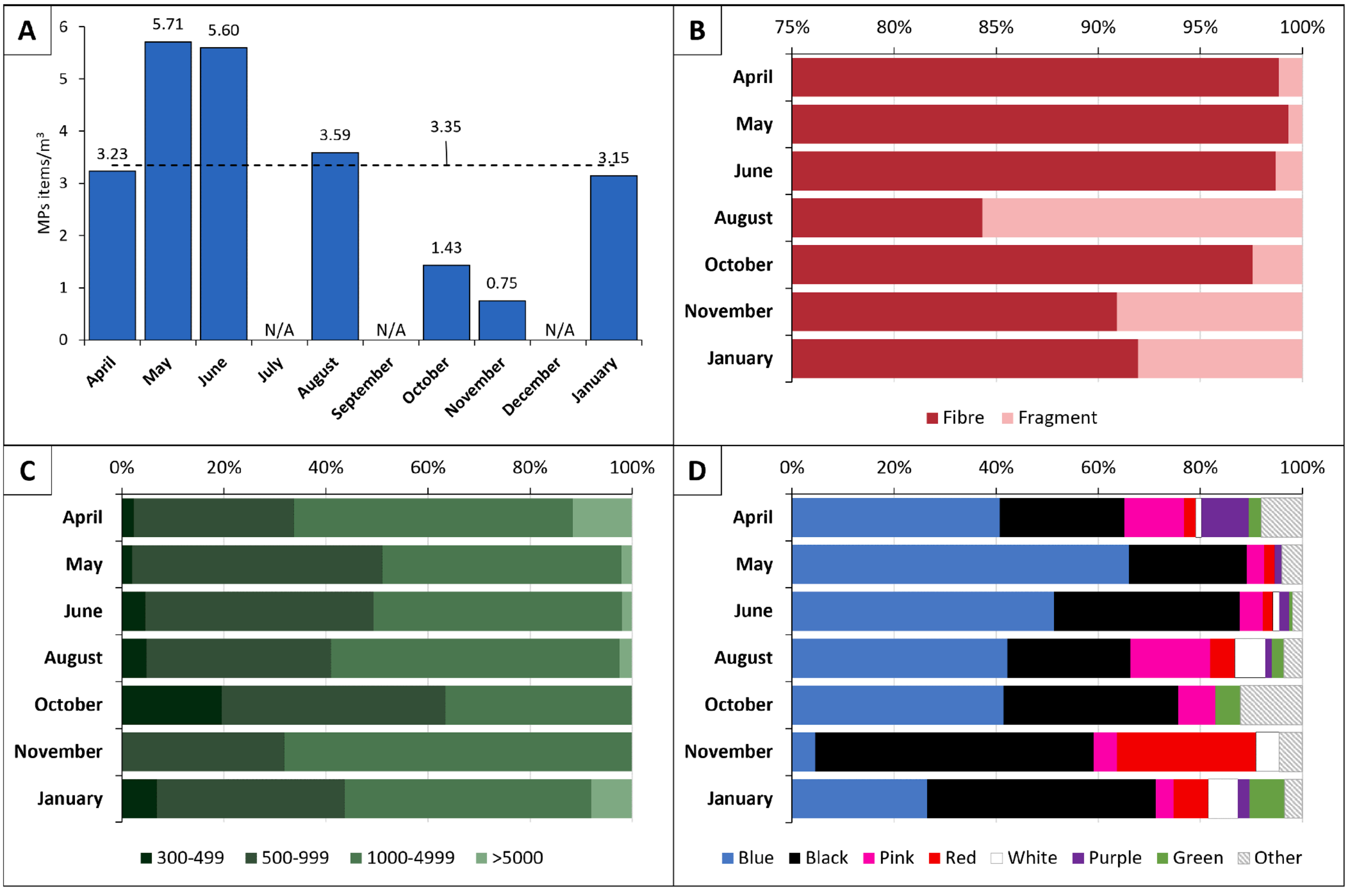

Fig. 3. Descriptive visualisations of the microplastic (MP) particles >300 μm found in Lake Velnezers surface water during 2019 (April, May, June, August, October, November) and 2020 (January): (A) seasonal dynamics of MP concentration (particles/m^3), dashed line indicates annual average and N/A indicates months when MPs were not assessed; (B) shape; (C) MP particle major dimension length, μm; and (D) colour.

fragments made up a relatively small proportion (4.35%) (Fig. 3B). A small proportion of particles were in the size ranges of 300-499 (4.84%) and >5000 μm (4.03%), while the majority were in the size groups of 1000-4999 μm (50.00%) and 500-999 μm (41.13%) (Fig. 3C). The most common colours of the particles were blue (46.29%), black (31.61%), pink (6.77%) and red (3.87%), while other colours were less represented (Fig. 3D). Polymers accounted for only 3.06% of the total number of particles (n=19). On average, polypropylene (PP) constituted 59.89% of the analysed particles, followed by PE (15.79%), polyester (PES) and polystyrene (PS) (each 5.26%). A “hot needle” test was performed on 11.65% from the 96.94% of the particles not verified spectroscopically by ATR-FTIR, revealing that 57.14% of the tested particles were synthetic, while the remaining 42.86% of the particles were of non-synthetic origin.

3.5. Microplastic deposition in sediment traps

Over the one-year study period, the average MP deposition rate was 9.47 particles/cm^2/yr or 4.31 μg/cm^2/yr. During the autumn/winter season, MP deposition was greater (16.52 particles/cm^2/yr or 7.43 μg/cm^2/yr) than that in the spring/summer period (3.98 particles/cm^2/yr or 1.88 μg/cm^2/yr) (Fig. 4). The most prevalent shape of the settled particles was fragments (99.54%), and fibres were found only in the spring/summer sample. However, there was a relatively higher amount of more elongated particles (AR 0<0.25 and 0.25≤AR<0.5), accounting for 16.65% and 47.06% in the spring/summer sample and 5.54% and 56.36% in the autumn/winter sample, respectively, rather than rounder ones (AR 0.5≤AR<0.75 and 0.75≤AR≤1). The dominant particle size classes corresponding to the major dimensions were 100-199 μm (53.24%), 50-99 μm (21.30%) and 200-299 μm (14.81%) for both

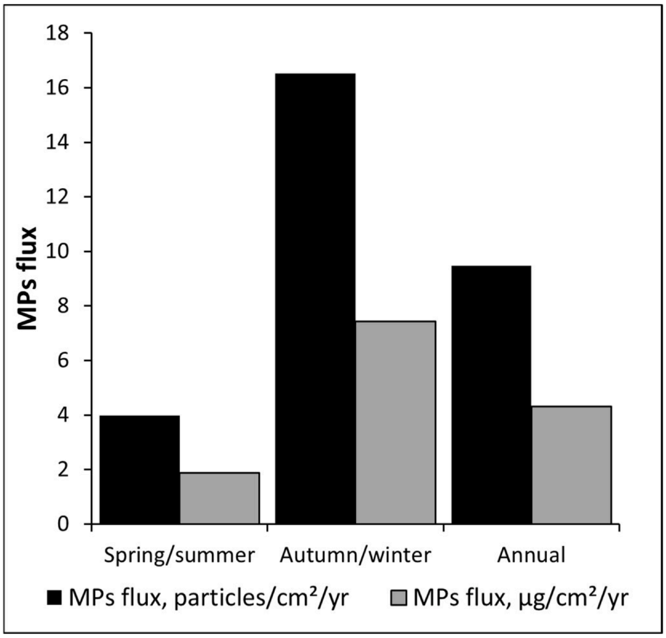

Fig. 4. The accumulation rates of microplastic (MP) <500 μm (particles/cm^2/yr and μg/cm^2/yr, left axis) and sediment (mg/cm^2/yr, right axis) in Lake Velnezers.

seasons. Some particles were found outside the defined highest (500 μm) size range. The identified polymers were PE (57.87%), PP (25.00%), PS (15.28%) and PA (1.85%). The autumn/winter sample contained more PE than PP; however, the spring/summer sample was dominated by PP.

3.6. *Microplastics throughout the sediment core*

MPs were detected throughout the sediment core in concentrations varying from 5.20 MP particles per gram of dry sediment (or 4.43 μg/g) in the deepest sediment layer representing years prior to 1890 (30-32 cm) to 129.00 particles/g (or 71.97 μg/g) in the layer from 2016-2018 (2-4 cm), with average concentrations of 43.96 ±34.69 particles/g (or 20.99 ±17.90 μg/g) per core layer (Fig. 5A). The highest MP concentrations were found in the uppermost sediment layers (0-8 cm), representing the period 2019-2009. Overall, a negative correlation ($r = -0.69$, $p = 0.003$, 95% CI [-0.884, -0.298]) was observed between sediment depth and MP concentration, indicating that MP concentration decreases as sediment depth increases (Suppl. Mat. Fig. S2A), despite the sediment layer at 20-22 cm (representing the years 1973-1966) having noticeably higher MPs pollution (95.19 particles/g or 47.00 μg/g) than adjacent layers. The bulk density of the sediments proved to be a statistically insignificant parameter in relation to the MP abundance in the respective sediment layer, with a weak negative correlation ($r = -0.31$, $p = 0.24$, 95% CI [-0.701, 0.215]) (Suppl. Fig. 2A). Similar to the trap samples, in all core samples, the dominant particle shape was fragment, constituting 97.93% on average (Fig. 5B). However, there was relatively higher amount of more elongated particles ($0.25 \le AR < 0.5$, $AR\ 0.5 \le AR < 0.75$ and $AR\ 0 < 0.25$) rather than rounder particles ($0.75 \le AR \le 1$) (Fig. 5C). The proportional differences of the defined AR groups in each sediment layer did not reveal a correlation between particle elongation and depth (Suppl. Mat. Fig. S2B).

In terms of the major dimensions of the particles, the 100-199 μm size group was dominant (49.18%), followed by the 200-299 μm (22.57%) and 50-99 μm (16.25%) size classes (Fig. 5D); some particles were found outside the defined largest size range (1.96%). The most abundant polymers in the sediment core were PS, PE and PP, making up 38.28, 38.17 and 16.47% of the total polymers, respectively (Fig. 5E). The proportions of PA, acrylonitrile butadiene styrene (ABS) and polyurethane (PU) were lower – 6.11, 0.65 and 0.33%, respectively. PU was found in small amounts only in layers 6-8 and 14-16, representing 2013-2009 and 1991-1985, respectively.

4. Discussion

4.1. *Quantification and seasonal occurrence of MPs in lake surface water*

The use of surface water MP samples is most straightforward in regard to sampling, purifying and analysis (Pasquier et al., 2022). The surface water MP concentrations reported here (0.75-5.71 particles/m^3) are on the same order of magnitude as those reported in comparable studies, i.e., in Lake Winnipeg, Canada, surface water samples contained 0.58-8.31 particles/m^3 (Anderson et al., 2017), and in Lakes Chusi and Bolsena, Italy, 2.68-3.36 and 0.82-4.41 particles/m^3, respectively (Fischer et al., 2016). Lower MP pollution was reported by Uurasjärvi et al. (2020) from Lake Kallavesi, Finland (0.037-0.66 particles/m^3), and by Sighicelli et al. (2018) describing MP pollution in three Italian subalpine lakes (0.02-0.28 particles/m^3). A study investigating MP pollution in Lake Lugano (Switzerland, Italy) applying similar sampling

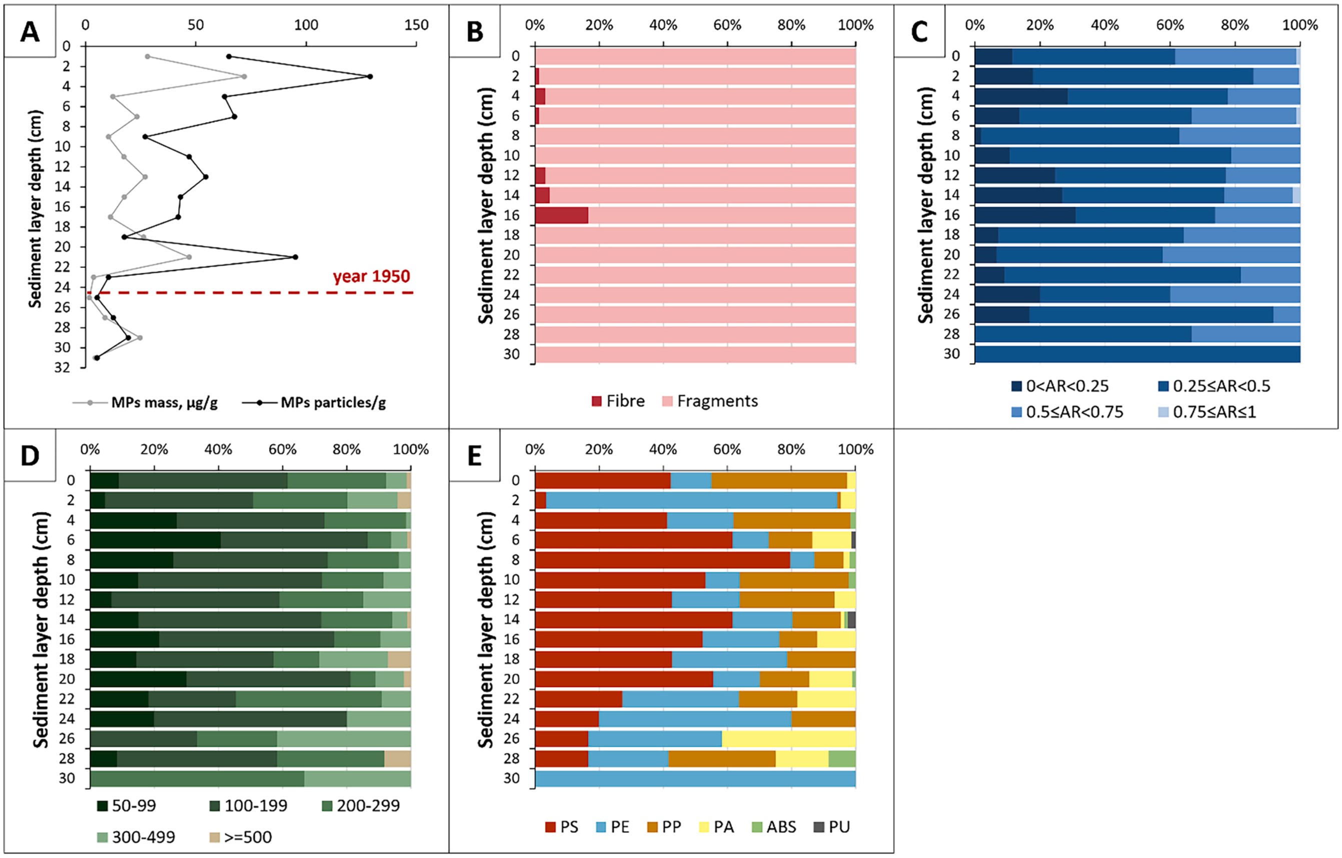

Fig. 5. Descriptive visualisations of the microplastic (MP) particles found in the Lake Velnezers sediment core: (A) MP concentration (particles/g) and mass (μg/g), the red dashed line indicates the sediment layer representing the year 1950 when plastic mass production was assumed to have begun; (B) shape; (C) aspect ratio (AR); (D) major dimension length of the MP particles, μm; (E) polymer type (PS – polystyrene, PE – polyethylene, PP – polypropylene, PA – polyacrylate, ABS – acrylonitrile butadiene styrene, PU – polyurethane).

method (net trawling, mesh size 250 μm) revealed greater MP pollution than the present study (11.5 particles/m^3) (Nava et al., 2023) and concluded that the size of the lake and the urbanisation level of the surrounding area are major contributors to MP pollution. The results reported by Su et al., 2016, from one of the largest lakes in China receiving growing urban and industrial pressure, Lake Taihu, are among the highest recorded freshwater lakes worldwide, with MP concentrations ranging from 3400 to 25800 particles/m^3. Lake Velnezers area has been highly urbanised and industrialised for the last century, and this environment might have played a major role in increasing the extent of MP pollution in the waterbody.

Considering seasonal occurrence, in our study, surface water samples from the late spring (May 2019) and early summer (June 2019) contained not only more MPs but also more organic matter, which is a prominent characteristic of eutrophic shallow lakes during growing season. This may have caused MPs to become trapped in floating microalgae and, therefore, increase total surface water MP pollution (Atugoda et al., 2020; Corcoran et al., 2015; Li et al., 2023a; Zhang et al., 2020). The seasonal aspect should also be considered when planning monitoring in regions where the amount of precipitation strongly varies seasonally (i.e., during the wet and dry seasons). For instance, the MP concentrations in Lake Phewa, Nepal, were significantly greater in the dry season (2960 particles/m^3) than in the rainy season (1510 particles/m^3), which is attributed to storm and rainfall events prior to the winter sampling (Malla-Pradhan et al., 2022).

The disadvantage of surface water sampling for MP monitoring is the high dependency on meteorological factors (Fischer et al., 2016) and limited availability of different density and shape polymers (Atugoda et al., 2020; Hidalgo-Ruz et al., 2012; Kowalski et al., 2016; Kumar et al., 2021). For instance, fibres were the most common particle shape in most of the mentioned studies (Anderson et al., 2017; Malla-Pradhan et al., 2023, 2022; Su et al., 2016; Uurasjärvi et al., 2020), similar to our survey. The most frequent polymers found in current and other studies were PP and PE (Malla-Pradhan et al., 2023; Sighicelli et al., 2018b; Uurasjärvi et al., 2020); however, it is important to note that only a relatively small portion of the particles found in surface water were subjected to chemical analysis (3.06%) in the present study, and the results could differ if more particles would be assessed. Nevertheless, of the 11% of randomly chosen particles in our study subjected to the "hot needle" test, close to half (42.86%) were of non-synthetic origin. The dominance of non-synthetic over synthetic MP fibres from freshwater and the atmosphere has been shown by Stanton et al., 2019, while the dominance of non-synthetic MPs from oceanic surfaces has been shown by Suaria et al., 2020. Both studies concluded that most (more than 90%) of floating fibres are not MPs but rather natural, i.e., of animal or plant origin. If the results supported by optical microscopy are not verified by chemical characterisation, this may lead to misinterpretation of the degree of pollution.

4.2. Quantification of MPs in dated sediment cores

The concentrations of surface sediment MPs can vary, but the application of the coring method provides a better snapshot of the historical retention of MPs in sediments (Uddin et al., 2021). MP deposits in sediments have even been suggested to serve as markers of the Anthropocene since the starting point of this unofficial epoch coincides with the beginning of plastic mass production (Zalasiewicz et al., 2016). The following challenges, however, exist: sediment cores are more challenging to sample, and material might be mixed; therefore, it will not be possible to estimate the actual corresponding time versus pollution. Even when well-dated sediments are present, considerable amounts of MP pollution can still be found in sediments that accumulated before the Anthropocene (Dimante-Deimantovica et al., 2024; Martin et al., 2022). Our findings also support this observation, confirming that MPs were found in sediment layers representing time well prior to the beginning of plastic mass production. Based on the results of the positive and negative controls, we excluded the possibility that the occurrence of MPs in sediments older than 1950 was caused by procedural contamination. The presence of MPs deeper than the layer representing 1950ies indicate MPs migration deeper in the sediments, potentially due to sediment compaction, the physical characteristics of the particles or sediments, or biological processes such as bioturbation (Dimante-Deimantovica et al., 2024; Gebhardt and Forster, 2018; Huang et al., 2022). In the present study, MP abundance showed an upwards trend from the bottom (prior to years 1890) to the surface (years up to 2019), a feature characteristic of similar studies of MPs in sediments (Dimante-Deimantovica et al., 2024; Li et al., 2023b; Martin et al., 2022). In contrast, there was no clear trend toward a gradual increase in MP abundance in the sediment core of Lake Taihu, China (Huang et al., 2022). Considerably fewer MPs were found in the sediments of Lake Jianhu, China (0.13-2.94, on average 0.92 particles/g) (Li et al., 2023b) than in those of Lake Velnezers (6.93-138.00, on average 49.53 particles/g). However, the size of the found MPs was greater in Lake Jianhu than in Velnezers, potentially indicating that particle size is correlated with the abundance of MPs. The most common MP polymers in Lake Velnezers are PE, PS and PP. In addition to the abovementioned polymers, other studies have also identified higher abundances of PA, PET, rayon, rubber and other polymers, which are found in Lake Velnezers at much lower concentrations or are not detected at all (Dimante-Deimantovica et al., 2024; Huang et al., 2022; Li et al., 2023b).

4.3. Quantification and seasonal occurrence of MPs in sediment traps

Despite being widely used for decades in geology studies (Bloesch and Burns, 1980; Blomqvist and Håkanson, 1981), sediment traps have only recently been considered a powerful tool for the assessment of MP accumulation (Saarni et al., 2023, 2021). Measuring sinking MPs is the most direct method since traps collect material over a limited period of time (i.e., how many MP particles are accumulated per area per time unit), covering the whole vertical water profile. This approach enables intercomparability free from the influence of varying sedimentation rates, which can hinder comparisons relying on MP concentrations. We found that the autumn/winter season sample contained more MPs than the spring/summer season sample, which might be related to greater sedimentation of natural particles, such as algae, fallen tree leaves and aggregates, during this time of the year (Dai et al., 2018). Our results contrast with those of a study carried out in an urban lake in Finland that reported a lower accumulation of MPs during the winter season than during the productive season (Saarni et al., 2021). However, the study mentioned above observed almost seven times lower annual MPs flux rate (3.24 particles/cm^2/yr), than the present study (22.20 particles/cm^2/yr). The study of Arkona Basin, Baltic Sea, reported the annual MP flux to be significantly less prominent (0.0037 particles/cm^2/yr) (Enders et al., 2019), similar as in Lake Kallavesi, Finland, where the annual MP flux varied from 0.07 to 0.23 particles/cm^2/yr (Saarni et al., 2023). The characteristics of the MPs are generally consistent with those reported in other studies, revealing that most of the particles were fragments, followed by fibres (Saarni et al., 2023, 2021). The most frequent polymers found in current and other studies were PE and PP in different proportions (Saarni et al., 2021, 2023).

4.4. Relationships between the studied matrices

The fate of most MPs, also with low polymer densities, in aquatic systems leads to their vertical sinking from the water surface through the water column, ultimately leading to their accumulation in sediments due to factors such as density-driven sinking, particle biofouling, and heteroaggregation (Atugoda et al., 2020; Dai et al., 2018; Forrest et al., 2020; Horton and Dixon, 2018; Li et al., 2023a; Nayebi et al., 2023; Shamskhany et al., 2021). We examined whether a relationship between MP characteristics in the studied compartments could be observed.

Surface water and sediment traps. A comparison between tendencies in the abundance of MPs in surface water and the amount of deposited MPs in the corresponding time period revealed a delay in the seasonal abundance of MPs; surface water MP concentrations were greater in the spring/summer (April through August) season and lower in the autumn/winter season (September through January), while in the trap samples, it was the other way around. Higher MP concentrations in traps during the autumn/winter season may occur due to seasonal variations, e.g., relatively lower primary production, colder and less turbid water, and increased vertical downwards transport of suspended matter (Horton and Dixon, 2018; Wagner et al., 2014). Surface water may in turn contain particles transported from catchment area (Dusaucy et al., 2021) into lakes during spring/summer when the MP concentration on the lake surface is greater than that in autumn/winter. Seasonal algae blooms can trap MP particles (Atugoda et al., 2020; Corcoran et al., 2015; Li et al., 2023a; Zhang et al., 2020), ensuring their occurrence in surface water and the water column and enabling sedimentation (i.e., accumulation in sediment traps) towards the end of the productive season. One may also expect more particles to sink after snow and ice melt in spring, as snow is known to be a deposition matrix for airborne MPs (Bergmann et al., 2019). This, however, would not be the case of our study in particular case, since samples representing spring/summer (including melting period) were acquired during the year 2019 – the warmest year in the observation history (since 1924) with no snow cover. Also, the annual precipitation was 9% below the annual norm (692.3 mm), with the month of April marking driest and the month of November – the second most rainfall-rich months in the history of Latvia's meteorological observations (LVĢMC, 2020).

The surface water samples contained proportionally more fibres than the sediment traps, suggesting that shape might be an important factor promoting particle sinking and deposition (Atugoda et al., 2020; Kaiser et al., 2017; Li et al., 2023b). Particles with larger surface areas, such as fibres, tend to reside in a suspended state in water for a longer time period than fragments due to water turbulence and are subjected to horizontal transport to a greater degree (Atugoda et al., 2020; Kumar et al., 2021). A slow lake water flow velocity enhances MP sedimentation processes, especially in small lakes such as the Velnezers (Lambert and Wagner, 2018). Additionally, it is known that synthetic particles tend to break down over time due to mechanical, chemical or biological degradation (Mitrano et al., 2021; Sipe et al., 2022); therefore, it is conceivable that fibre-like particles fragment into smaller pieces, acquiring the dimensions of fragments over time. This might be the reason why the current study revealed the greatest percentage of fibres in surface water and fragments in sediment trap and core. Additionally, non-synthetic origin fibre particles (representing nearly half of all surface water particles in our study) may not accumulate in sediments due to decomposition.

Sediment traps and core. The sediment core chronology suggested a surface mixing zone at a depth range of 0-8 cm (age range 2019-2009); therefore, the actual MP concentrations might not be directly accurate in these chronology-bound layers. However, they can be used as integrated samples for comparison with MPs deposited in sediment traps over a one-year period based on the following criteria: shape, elongation, size and polymer.

The proportions of particle characteristics in the sediment trap samples and upper layers of the core corresponded. The unequivocally dominant particle shape was fragments (Fig. 6A), and most of the particles were rectangular with ARs of $0.25 \leq AR < 0.5$ and $0.5 \leq AR < 0.75$ (54.17 and 36.11% for the trap samples and 60.00 and 21.88% for the sediment core samples, respectively), while in both studied compartments, there was the least number of roundish particles ($0.75 \leq AR \leq 1$) (Fig. 6B). Additionally, the most represented size groups were common for both matrices, with 100-199 μm being the most abundant, followed by the 50-99 μm group for the trap samples and the 200-299 μm group for the core samples (Fig. 6C). Most of the particles in both matrices were composed of PE, PS and PP (Fig. 6D).

4.5. *Which matrix should be chosen for lake microplastic pollution monitoring?*

The abiotic environmental compartment chosen to represent MP

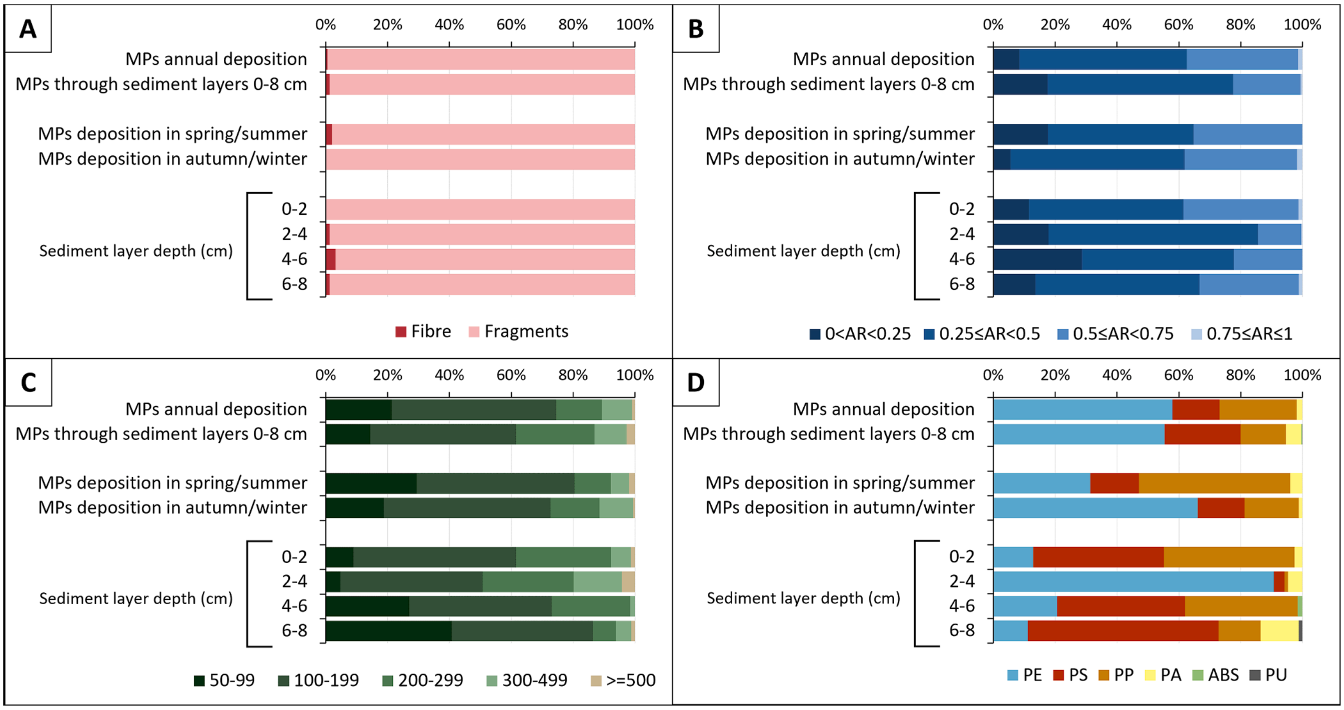

Fig. 6. Comparison of the microplastic (MP) particles found in Lake Velnezers sediment trap samples and upper layers (0-8 cm) of the sediment core representing (A) shape; (B) aspect ratio (AR); (C) major dimension length of MP particles, μm; and (D) polymer type (PE – polyethylene, PS – polystyrene, PP – polypropylene, PA – polyacrylate, ABS – acrylonitrile butadiene styrene, PU – polyurethane).

pollution in lakes should be able to provide information on current-time pollution levels, show temporal trends, provide reliable and comparable data, be cost- and time-efficient throughout the assessment process, and reflect the environmental risk of MPs in lake ecosystem (Chen et al., 2024; Lu et al., 2021). Nevertheless, each of the tested matrices (surface water, sediment core and sediment trap samples) presents some challenges and advances in MP pollution monitoring in lakes.

The collection, processing and analysis of surface water MP samples are relatively simple, quick and, therefore, inexpensive, offering great spatial coverage. However, drawbacks related to the use of this matrix for monitoring purposes include, e.g., particles consisting mostly of polymers with a density lower than that of water being present in the surface water, and the dependence of the MP concentration on meteorological and hydrophysical conditions (Fischer et al., 2016), which can vary within a few hours. This, in turn, can be solved by performing replicate sampling. Besides, the lowest size border using Manta trawl is dependent on the productivity of the lake, i.e., the application of mesh size smaller than 300 µm might cause net clogging and reduce the ability to filter representable water volume in eutrophic lakes, leaving out smaller size MPs particles. Therefore, while the application of 300 µm mesh might lead to underestimation of the actual pollution level, it offers a feasible method for regularly monitoring MP pollution changes in eutrophic lakes. If possible, pump systems with lower filtering mesh sizes are encouraged to acquire a more accurate view of actual MP pollution at smaller size range.

Non-disturbed sediment records enable the investigation of past pollution rates (Hinata et al., 2023) and potential downwards particle migration in sediments; however, these records do not reveal short-term changes in MP flux, and spatial coverage is limited to the accumulation part of the lake (United Nations Environment Programme, 2020). Sediment MP sample processing and analyses are among the most time-consuming and expensive methods. Intercomparability between different sites and locations is only possible with accurate and precise dating of the cores, which adds to the cost and time requirements. The number of MPs in sediment is expected to be less variable over time than that in water-based matrices; therefore, a high temporal resolution of sampling is not necessary.

The sediment trap approach is innovative in the field of MP research and provides information on the deposition of MPs over a certain real-time period, and the MPs are at lower risk of being affected by factors such as low-scale weather changes or bioturbation. Moreover, sediment traps show not only the amount of deposited MPs but also the amount of pollution that is potentially bioavailable to pelagic organisms in the water column. MP extraction from sediment traps is simpler than that from sediment core samples and does not require extensive treatment. Nevertheless, trap system installation and finding afterwards might require foreseen logistics and skills; additionally, the volume of collected sample might not be sufficient in lakes with very low MP fluxes in a short time – in this case, if high-resolution seasonal data are not needed, the trap system can be left for a longer time period.

There are, however, several ways to improve the methodology used in our study. For example, introducing samples replicas during surface water sampling to acquire more representative results. Investigation of MPs throughout water column at different depths would provide insights on sinking patterns and impact of water conditions, e.g., thermocline, across seasons. Impact of stratification and water mixing regimes in lakes should be studied more closely, as in seawater it is known the water column can contain more MPs compared to surface water (Uurasjärvi et al., 2021). Also, sediment traps should be investigated at different depths for the same reason. In addition to Manta trawling, water compartment MPs could be sampled using filtration device with smaller mesh size (10-50 um) so the results would be comparable to sediments. The study also needs to be repeated for a longer time period that would allow to test the feasibility of suggested matrices for long term MPs monitoring in lakes – e.g. surface water and sediment trap samples collected monthly to acquire higher temporal resolution and define most representative season or sampling duration. Although we looked at three important lake ecosystem matrices, assessing MPs in biota would provide valuable information on biological significance of MPs pollution in lake, which was not assessed in present study.

5. Conclusions

In this study, we quantified and holistically described microplastic (MP) pollution in small lake surface water, historical sediment records, and deposited MPs throughout a seasonally resolved one-year study period. Our findings revealed that none of the acquired samples were free of plastics, indicating the pervasive presence of this anthropogenic pollutant in the lake ecosystem, even in sediment layers dated before the onset of plastic mass production. Seasonal variations in MP concentrations were observed in both surface water and sediment traps, with a delayed relationship between the two compartments during different parts of the year. This highlights the dynamic nature of MP pollution in lakes and underscores the importance of considering temporal trends in monitoring efforts.

Combining multiple methods can provide a more comprehensive understanding of the presence and dynamics of MP pollution in aquatic environments. Hence, based on the current study and previous experience, we suggest advancing the combination of surface water net trawling and sediment trapping as primary abiotic lake compartments for MP monitoring purposes. This combination will ensure greater acquired data credibility, the possibility of acquiring information on current pollution levels, and the ability to assess temporal changes in current-time pollution. While Manta net trawling is a widely used and accepted method in both marine and freshwater environments, the sediment trap approach is a novel and efficient tool for monitoring MP accumulation rates in aquatic environments, providing an opportunity to better understand and define processes controlling MP accumulation. Both mentioned methods provide shorter, simpler, and less expensive treatment processes.

Future research should focus on evaluating long-term trends in MP pollution and assessing the feasibility of the monitoring strategies proposed in this study. Additionally, further investigations into MP vertical transport patterns, distribution throughout the water column and factors governing it, deposition mechanisms, and factors influencing sediment-water exchange processes will enhance our understanding of MP dynamics in lake systems. To fully assess MP contamination in freshwater environments, it is recommended to examine MPs in surface water, sediment traps, and biota simultaneously.

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CRediT authorship contribution statement

Marta Barone: Writing – review & editing, Writing – original draft, Visualization, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Inta Dimante-Deimantovica:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Sintija Busmane:** Investigation. **Arto Koistinen:** Writing – review & editing, Investigation, Formal analysis. **Rita Poikane:** Writing – review & editing, Supervision. **Saija Saarni:** Writing – review & editing. **Normunds Stivrins:** Writing – review & editing, Investigation, Funding acquisition, Formal analysis. **Wojciech Tylmann:** Writing – review & editing, Investigation, Formal analysis. **Emilia Uurasjärvi:** Investigation, Formal analysis. **Arturs Viksna:** Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Supplementary materials

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