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# Tracking the microplastic accumulation from past to present in the freshwater ecosystems: A case study in Susurluk Basin, Turkey

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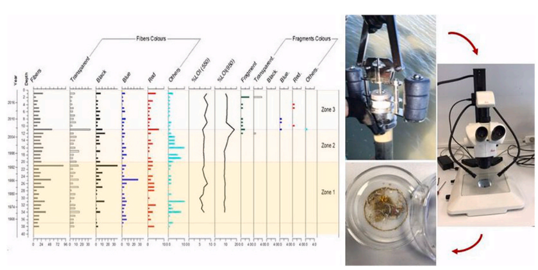
## HIGHLIGHTS

- Microplastics are a useful tool for paleolimnological studies revealing historical flux.
- Fibers were the dominant microplastic particles from the core samples.
- Rivers play an important role in transporting the microplastics in aquatic ecosystems.
- Conservation status of a freshwater system reduces the microplastic contamination.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Microplastic pollution in aquatic ecosystems has become a global issue in recent years due to its presence everywhere around the world. Although several studies have explored the impact of the accumulation of those small particles in marine environments, comparisons of freshwater systems with marine environments are scarce. In the current study, due to the lack of long-term data on microplastic pollution, we used paleolimnological approaches to acquire the missing information regarding this hot topic. Two short cores were taken from Bursa province in Turkey, which is the center of industrial and agricultural production with many different sectors such as textile and manufacturing. The first core sample was taken from a relatively pristine environment, Lake Uluabat, and the second one was taken from a delta area where all the discharge coming from the basin flowed through to the Marmara Sea. The sediment core from the lake was dated back to the 1960's and the majority of the sample was dominated by fibers. Despite there being no uniform distribution pattern, the number of the microplastics showed decreasing trend after the lake became a Ramsar site. Due to the continuous mixing in the sampling area, there were obstacles via the dating of the Delta core. Nevertheless, the data showed that a high number and variety of microplastics have accumulated over the last decade in the province. This can be interpreted as microplastic pollution reaching the sea directly from the basin. These findings revealed that a plastic chronostratigraphy would give important temporal data regarding the microplastic accumulation in aquatic ecosystems.

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## 1. Introduction

Since the first fully synthetic polymer *Bakelite* was produced in 1907, plastic production has been continuously growing worldwide due to its high durability, flexibility, lightness, ease of processing, disposability/reusability and affordability (Cózar et al., 2014; Patchaiyappan et al., 2020; Bertoldi et al., 2021). Plastic production currently amounts to 368 million metric tons (Statista, 2021) and is significantly increasing each year. The mass production, misuse, ineffective waste management and recycling of plastics contribute to the alarming increase in plastic debris in aquatic ecosystems (De Sá et al., 2018; Du et al., 2021).

Microplastics refers to plastic debris smaller than 5 mm in size (Fendall and Sewell, 2009; Hidalgo-Ruz et al., 2012). Different types of microplastics such as fragments, fibers, pellets, styrofoam, and film are found in aquatic ecosystems. They mostly originate from either primary microplastics – which are industrially produced as abrasives, exfoliators, textile microfibers and pre-production resin – or secondary microplastics resulting from the fragmentation of larger plastic wastes (e.g., single-use water bottles, plastic bags, fishing nets, etc.). Several different processes like photodegradation, physical scuffing and microbial effects are responsible for the degradation of the microplastics in different environments (Andrady, 2011; Zettler et al., 2013). Subsequently, these particles are transported by sewage treatment plants, atmospheric deposition, soil erosion or surface runoff to aquatic ecosystems (Browne, 2015; Horton et al., 2017a; Amrutha and Warrier, 2020; Hitchcock, 2020). Each year between 4.8 and 12.7 million tons of plastic enter the oceans via freshwaters, particularly as plastic waste discharges from rivers and lakes during extreme floods, so freshwater ecosystems play an important role in the plastic transport cycle (Bläsing and Amelung, 2018; Bertoldi et al., 2021).

The evaluation of microplastic accumulation in sediments is very crucial due to the sinking of toxic chemicals like trace metals, heavy metals, persistent organic pollutants (POPs), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and organochlorine pesticides that have the ability to attach to the microplastic surfaces and consequently might cause and enhance adverse health problems on aquatic organisms (Rios et al., 2007; Ren et al., 2020). Plastics are mostly made of polymers derived from non-renewable crude oil (Lithner et al., 2011) and are nearly indestructible. Moreover, the bioaccumulation of microplastics in the sediment can cause an increase in their percentage by biofouling. Therefore, when organisms feed on the sediment surface where microplastic accumulation has occurred, those particles can be transferred into the food chain and there is a higher chance of reaching upper-level organisms (Mato et al., 2001; Teuten et al., 2009; Cole et al., 2011; Rochman et al., 2014; Scherer et al., 2018; Aytan et al., 2022). This can cause adverse health effects on organisms exposed to microplastics (Lusher et al., 2013; De Sá et al., 2018; Marn et al., 2020; Miller et al., 2020). Although microplastic pollution is a relatively new topic, it can be considered an emerging concern as a contaminant in the environment (Wagner and Lambert, 2018).

The investigation of plastic accumulation in freshwater ecosystems is relatively new in comparison to marine ecosystems. To the best of our knowledge, there is no long-term plastic waste observation system (Barnes et al., 2009; Wagner et al., 2014; Li et al., 2020a). Due to the high persistency of plastics and their significant accumulation rates in all kinds of environments, it is crucial to evaluate the frequency of their occurrences and types over time. Based on their deposition contexts, lake ecosystems provide excellent data representing catchment scale sinks for microplastic debris. Researchers have used lake sediments to explore the changes in community ecology, ecological responses and evolutionary ecology via the deposition of fossils (Burge et al., 2017). There are also several studies revealing past environmental changes using different biotic proxies such as diatoms and sub-fossil cladocerans in Turkish lakes (Çakıroğlu et al., 2014; Levi et al., 2016). Lake sediment preserves physical and biogeochemical signals, thereby enabling the investigation of the lake and landscape evolution, environmental changes, and responses to ecological drivers (Sayer et al., 2010; Burge et al., 2017). Due to the measurable accumulation rates and preservation of different proxies, sediment records can reconstruct ecosystem changes at a higher temporal resolution (Last and Smol, 2001; Cohen, 2003). Dating techniques and stratigraphic methods can be used to develop a depth-date relationship in cores (Appleby, 2002). This allows the interpretation of stressors in historical deposition (Sayer et al., 2010; Smol, 2010; Korosi et al., 2013). In many places, paleolimnological perspectives have been applied to the investigation of temporal trends for trace metals, organic chemicals, pharmaceuticals, etc. There have also been some studies on microplastic accumulation on timescale but still very limited (Bancone et al., 2020). Microplastics in sediment layers have not been widely used to reflect annual deposition. In this context, using microplastic particles would be a new proxy for tracking the microplastic accumulation rates for a *ca.* 150 year period. Due to the lack of long-term data on microplastic accumulation and pollution, historical records can provide crucial information for both researchers and authorities to predict future trends of microplastics from a conservation perspective. The occurrence of microplastics in surface water or sediment from lakes have been reported in several papers (e.g. Free et al., 2014; Fischer et al., 2016; Lenaker et al., 2019; Bertoldi et al., 2021), but considering the chronological techniques, there are relatively few studies according to our Web of Science search (Matsuguma et al., 2017; Fan et al., 2019; Frei et al., 2019; Turner et al., 2019; Dong et al., 2020; Martin et al., 2020; Xue et al., 2020; Courtene Jones et al., 2020; Chen et al., 2020; Lin et al., 2020; Belivermiş et al., 2021; Uddin et al., 2021). Most of these studies were performed using deep-sea sediment, which is in line with the general microplastic research in marine systems (e.g. Belivermiş et al., 2021; Chen et al., 2020; Courtene-Jones et al., 2020; Lin et al., 2020). Few of them evaluated microplastic accumulation over time for freshwater environments (e.g. Fan et al., 2019; Turner et al., 2019; Dong et al., 2020). We aimed to investigate plastic pollution in a protected area to understand the human impacts and recent changes by analyzing short sediment cores dated by $^{210}$Pb and $^{137}$Cs, which are the main radiometric methods for chronological approaches. Three questions were addressed in the current study; i) do sediment records reflect the microplastic accumulation? ii) do identified microplastics reflect the anthropogenic activities? iii) does the polymer composition show variation through the dated sediment? Consequently, paleolimnological approaches are extremely valuable for tracking several changes, such as pollution, in ecosystems over time.

## 2. Material methods

### 2.1. Study site

Bursa is the fourth most populated city in Turkey and the center of industrial and agricultural production with many different sectors such as textile, manufacturing, automotives, and agriculture (Teksoy et al., 2017). The proximity of sampling sites to the highly anthropogenic activities triggers the contribution of both industrial and domestic pollution in Lake Uluabat and Kocaçay Delta. Studies showed that the sampling sites, particularly Lake Uluabat, have had an increasing eutrophication trend since the beginning of the 20th century (Salihoğlu and Karaer, 2004; Reed et al., 2008; Katip et al., 2015). The water fluctuation in the lake has shown variations, especially during the low water level years when agricultural activities were increased (Yersiz et al., 2001). The main means of existence are fishing, husbandry, farming as well as boat trips for touristic purposes around the lake (Personal Communication).

To investigate the microplastic accumulation in the Susurluk River Basin, two sites were selected from the Bursa province of Turkey (Fig. 1, Table 1). The first site was Lake Uluabat (40.1489°N, 28.6148°E) with a mean annual depth of 2.5–3 m and covering an area of approximately 155 km$^2$ during the maximum water level times and 135 km$^2$ at the minimum water level state (Dalkiran et al., 2006). The major inlet of the lake is Mustafa Kemal Paşa stream (~112 m3/sec high flow in March; ~14.4 m3/sec low flow in August), which is fed by polluted water from industrial discharge, sewage effluents, agricultural waste and waste from mining activities in the vicinity, and it drains into Kocacay River through the northwest (Degirminci et al., 2006; Katip et al., 2015). Due to its rich biodiversity in terms of aquatic plants and its being a prime location for migratory bird routes (Salihoğlu and Karaer, 2004) and freshwater sources, the lake was designated with Ramsar status in 1998.

The second sampling site was Kocacay Delta, which is 21 km long and 3.5 km at its widest (Irtem and Saçin, 2012) and discharges into the Marmara Sea from the south (Fig. 1, Table 1). The main economic activities around the river are agriculture and fishing. Kocaçay river connects to Nilufer stream, which is the dominant source of wastewater pollution from the highly urbanized Bursa city, and Susurluk River – the main water source of the delta (Tavsanoğlu and Akbulut, 2019).

### 2.2. Coring and dating

Sediment samples were extruded from the deepest point of Lake Uluabat and the Kocacay Delta using a KC-Denmark Kajak Corer (32/20 short corer) in August 2020. Two core samples were taken at each site for dating and microplastic detection. The data of the deepest point were obtained from a bathymetric map by Kazancı et al. (2004) and Aksoy et al. (2016). The length of the cores was 38 cm from Lake Uluabat and 17 cm from Kocaçay Delta. The core samples were sliced into 1 cm thick layers using a stainless-steel blade and stored at +4 °C until laboratory analysis.

For each site, freeze-dried sediment samples were dated at the Gamma Dating Centre, Institute of Geography, University of Copenhagen. The chronology of the cores was calculated using a modified method of the constant rate of supply (CRS)-modeling (Appleby, 2002; Andersen, 2017). Sediments from both sites were analyzed for the activity of $^{210}$Pb, $^{226}$Ra and $^{137}$Cs via gamma spectrometry. The measurements were carried out on a Canberra low-background Germanium well-detector. $^{210}$Pb was measured via its gamma-peak at 46.5 keV, $^{226}$Ra via the granddaughter $^{210}$Pb (peaks at 295 and 352 keV), and $^{137}$Cs via its peak at 661 keV. Loss on ignition (LOI) analyses were also conducted for each layer to detect the organic and carbonic matter content of the samples (Heiri et al., 2001). The samples were weighed and dried overnight at 105 °C and placed inside a furnace for at least 2 h at 550 °C once the new weights were recorded. Thereafter, samples were heated to 950 °C for 2 h and placed in a desiccator before the re-weighing process. The necessary calculations were then made to maintain the organic and carbonic content of the sediments (Heiri et al., 2001). A C2 program was used to analyze core data and the production of a stratigraphic diagram of microplastic distribution along with the core samples (Juggins, 2007).

**Table 1**
General characteristics of study sites.

| Variables | Lake Uluabat | Kocaçay Delta |
|---|---|---|
| Temp (°C) | 22 | 24.6 |
| DO (%) | 84.7 | 90.1 |
| EC (μS cm$^{-1}$) | 607 | 24751.3 |
| Salinity (‰) | 0.31 | 14.9 |
| Chl a (mg L$^{-1}$) | 37.35 | 101.7 |
| Secchi Depth (cm) | 20 | 80 |
| Trophic Class[a] | Eutrophic | Hypertrophic |

[a] Calculated according to the chlorophyll value after Carlson 1996.

### 2.3. Current microplastic sampling

To investigate the current MP characteristics, we collected top 0–6 cm bottom sediments from the sampling site, which was very close to the coring area using a Van Veen Grab sampler. The sediment samples were collected in three replicates and pooled in a stainless-steel container. The sediments were mixed with a stainless-steel large spoon and later kept in aluminum foil wrapped glass containers. To investigate surface microplastic contamination, manta trawls with flow-meter (General Oceanics) attachment were also performed at standard speed on the same sampling date and sites. The volume was calculated according to the manual of General Oceanics flow and current meter. To avoid contamination, we thoroughly rinsed with distilled water in a stainless-steel collector. The residue was transferred to a glass jar and stored in a cooling box until further analysis in the laboratory.

### 2.4. Microplastic analysis

Thirty-eight (Uluabat) and seventeen (Delta) slices from the cores were used to evaluate the microplastic accumulation. We followed two extraction procedures according to Masura et al. (2015). In each sediment sample, 25 g wet weight was measured for each slice. The lake sediment has silt-dominated lithology (Kazancı et al., 2010); therefore,

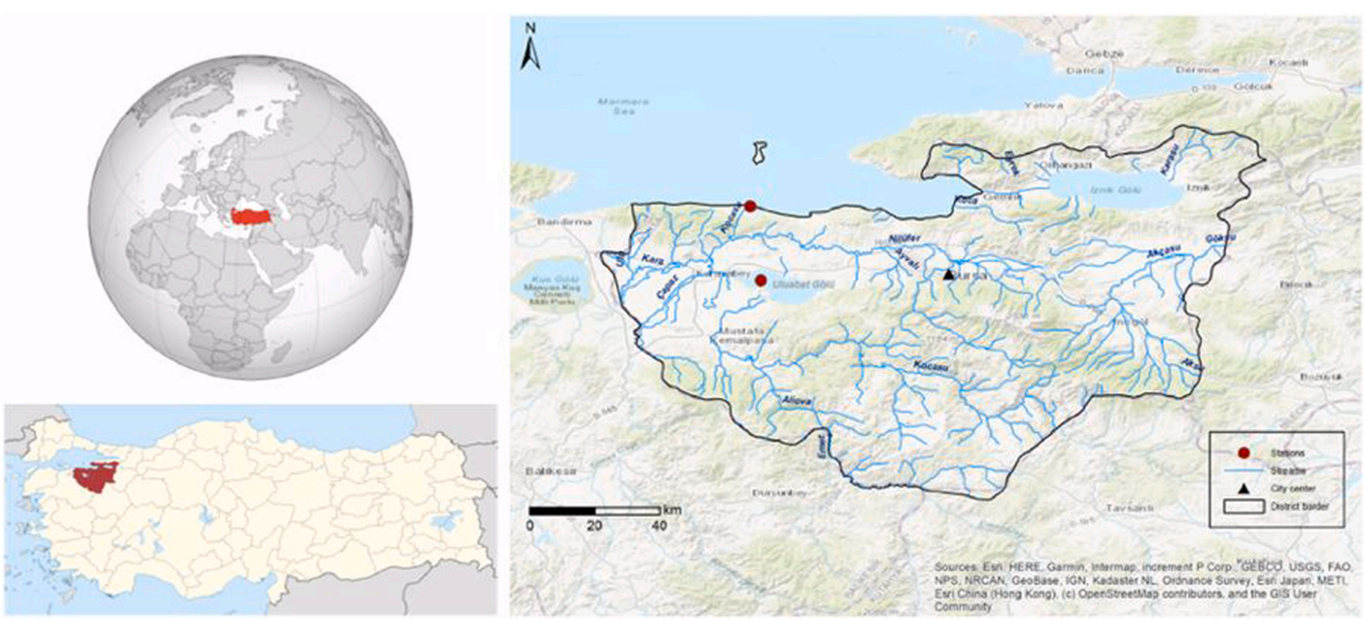


**Fig. 1.** Location of core sampling sites.

to improve extraction rates, the clayey mud content of the samples was removed by sieving through 0.5 mm, 0.2 mm, and 0.1 mm, respectively. (Esiukova et al., 2020). All remaining solids on the sieves were transferred to a beaker. For the Delta samples, which have larger grain size density, separation was applied. Due to the highly toxic or quite expensive properties of several salt solutions such as $ZnCI_2$, NaI, and sodium polytungstate (Torres and De-la-Torre, 2021), we preferred to use NaCl for density separation. Twenty-five grams of each sediment were put into a 250 ml glass beaker, and concentrated NaCl (1.2 g $mL^{-1}$) solution was added (Thompson et al., 2004; Hidalgo-Ruz et al., 2012). Sediments were stirred with a clean glass rod for 2 min and allowed to settle for 24 h (Wang et al., 2017; Peng et al., 2018). After 24 h, the supernatant of the sample in a beaker was removed carefully around 10 times (Belivermiş et al., 2021). During the removal of natural organic materials, all the samples were digested with Fenton's reagent (a mixture of 30% $H_2O_2$ and 0.05 M $FeSO_4$ .$7H_2O$) at 45 °C on a hot plate (Hurley et al., 2018; Lloret et al., 2021)

Subsequently, the samples were filtered through GF/C Whatman filters with a pore size of 1.2 μm under stainless steel vacuum filter. After filtration, filter papers were placed in clean glass Petri dishes and kept in the incubator for 24 h at 45 °C to avoid molding. Identification and counting of the samples were done under a Leica Flexacam C1 camera attachment M80 Stereomicroscope with LED illumination and with magnification at 17.5X- 60X. Different types of microplastics were identified visually according to their colors and types using needles, pens and tweezers (Hidalgo-Ruz et al., 2012). Fibers were observed for equal thickness throughout their length, those that branched out like organic hairs and plant fiber were not recorded as fibers, and films were checked for any cellular organs (Turner et al., 2019). To evaluate the size ranges of the identified microplastic, samples were photographed and measured. The assessment of sizes was carried out using an open-source scientific image-analysis program, ImageJ (Abramoff et al., 2004).

The chemical structure of polymeric microparticles was evaluated by FT-IR spectroscopy. A FT-IR spectrometer Tensor II (Bruker), equipped with ATR, was used to identify the polymer composition of microparticles. Spectra were obtained in ATR mode, with the resolution of 4 $cm^{-1}$ wavenumbers at 24 scans per sample and recorded in a range of 4000 to 400 $cm^{-1}$ wavenumber using OPUS (Bruker) vibrational spectroscopy software.

### *2.5. Quality assurance and quality control (QA/QC)*

To avoid microplastic contamination in the samples, we used clean metal or glass materials with the application of distilled water whenever possible during the core collection and slicing in the field. During the laboratory processing, cotton coats were worn and any unnecessary contact with plastic equipment was avoided. All washings were done with metal sieves and covered with a glass Petri lid when not in use. Prior to using a stainless-steel vacuum filter, we washed it using distilled water and covered it with aluminum foil to avoid airborne contamination until usage. All the organic matter removal processes were done in a fume hood. The blank sample was placed on the laboratory bench to compare the contamination. There were few (2–3) fibers observed and we exclude these from the total number. Prior to using commercial NaCl during density separation, we filtered the dissolved NaCl through GF/C Whatman filters with a pore size of 1.2 μm under a stainless-steel vacuum filter to avoid any contamination.

## 3. Results

### *3.1. Lake Uluabat*

A number of 919 fibers were observed in the kajak core samples from Lake Uluabat. From the whole 38 cm core sample, fibers of different colors were the dominant microplastic type. The most abundant colors were transparent (34%) black (27%), blue (19%), and red (8%). Other colors were also detected (such as yellow, pink, and green fibers) but in smaller amounts (10%) (Fig. 2). From the bottom to the top of the core, only 1.3% (12 items/total core) of the fragments were identified in the sediments after 2006 from Lake Uluabat (Fig. 3). Although no clear distribution pattern exists for film, there was only 0.5% (5 items/total) of film identified from the bottom to the top of the core. From the whole core, neither microbead nor foam was detected. The mean size of the microplastics was 1.38 ± 0.9 mm; however, no particular size distribution pattern was observed from the top to the bottom of the core sample. The size ranges of microplastics were 0.049–4.951 mm. A small representative group of microplastics (27 samples) in the core samples from Lake Uluabat was evaluated with FT-IR microscopy for chemical identification. However, only 4 of the samples were identified; 9 of them had no conclusive spectra and 14 of the sample spectra was not possible to interpret (SI Table 1). While identified samples were polypropylene, polyethylene terephthalate and polystyrene, the non-conclusive ones are polyethylene terephthalate and acrylic polymers in general. Nevertheless, size and type dependent limitations for microplastic identification resulting in no particular distribution through zones and depth of core. However, current microplastic samples (8 sediments and 6 water surface samples) from the same sampling point and season were used to reveal the polymer composition of microplastics with FT-IR spectroscopy with respect to the similarities between the core and surrounding environment. A similar spectral pattern was observed in the current water and sediment samples (SI Table 2). Representative selection of FT-IR spectra for the core and the current microplastic samples are given in SI Figs. 1 and 2.

Microplastic accumulation went on as far back as the mid 1960s (30–35 cm) according to the dating results on the deepest layer, while the topmost layer was dated to 2020 (Fig. 4). The sedimentation rate of Lake Uluabat was 2.52 kg $m^{-2}$ $y^{-1}$ on average at the deepest point.

Microplastics are distributed irregularly throughout whole core slices considering each depth/age interval. From 1966 to 1996 (37-20 cm), the microplastic accumulation showed an increasing trend over time (Fig. 4). Then, it peaked in the mid-1990s with the highest number of 90 particles. Since the end of the 1990s, the amount of microplastic

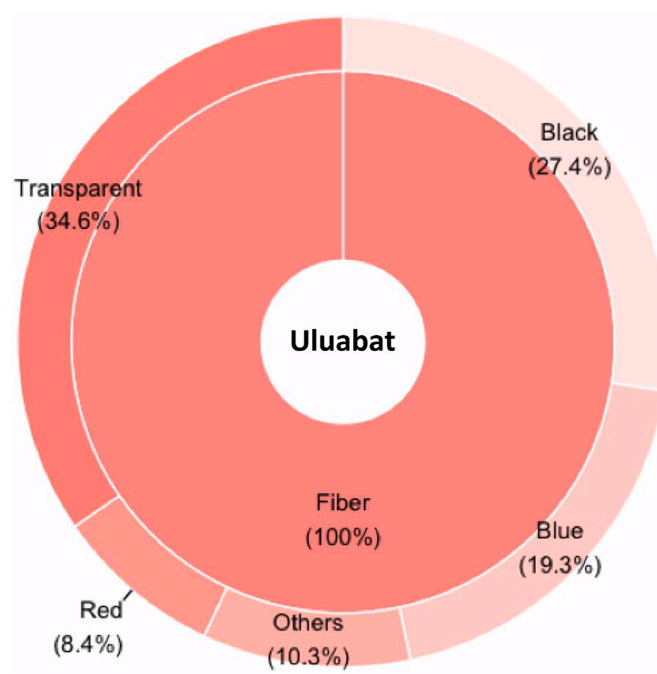


**Fig. 2.** Percent distribution of different colors of observed fibers from Kajak Corer in Lake Uluabat. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

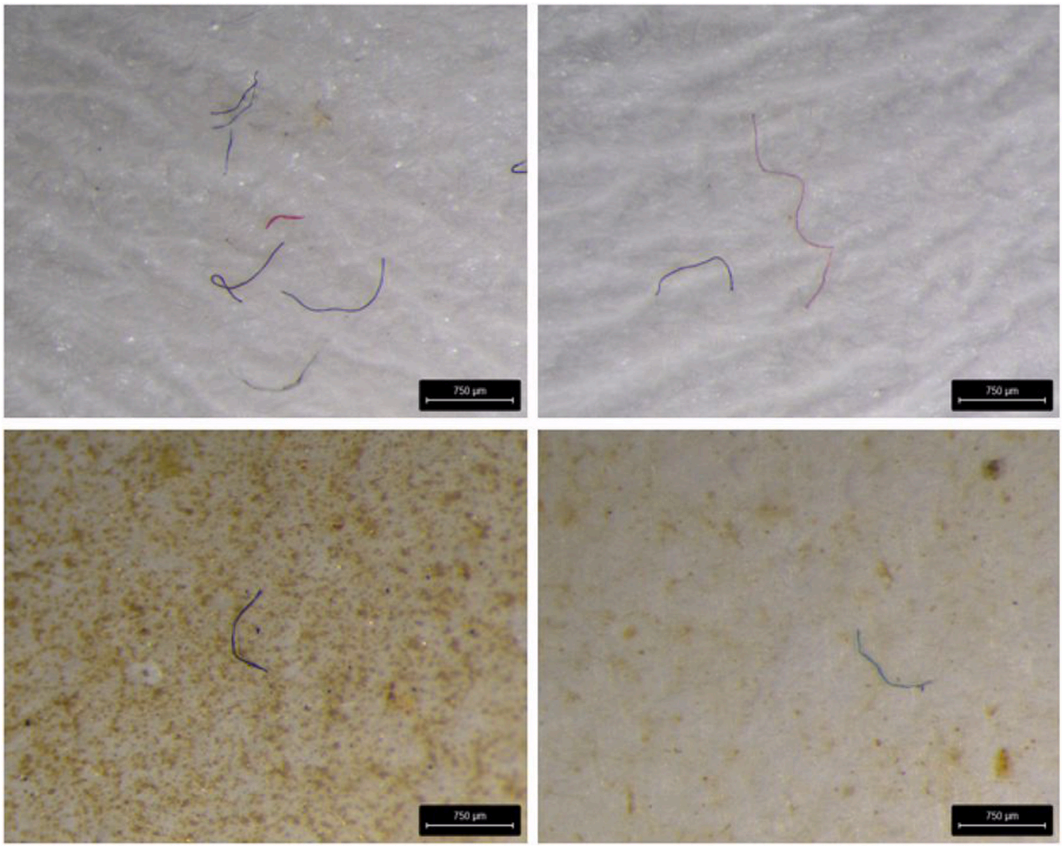


**Fig. 3.** Identified fibers from sediment core samples from Lake Uluabat.

**Table 2**
Distribution of microplastics through 2015 to 2020 in Kocaçay Delta.

| Types | Transparent | Black | Blue | Red | Yellow | Green | White | Total |
|---|---|---|---|---|---|---|---|---|
| Fiber | 92 | 498 | 338 | 307 | 46 | 40 | – | **1297** |
| Fragment | 20 | 10 | 42 | 13 | 5 | 13 | 16 | **118** |
| Film | 32 | 3 | 14 | 2 | 1 | 1 | 19 | **72** |

accumulation has decreased except for the year 2007, when the amount detected was 56 particles/zone. At the top of the core, the fibers relatively increased to around 29 particles/zone. From the current sediment samples taken by Van Veen grab, we detected 276 items/m$^2$. Approximately 64% of them were fibers and the rest of the particles were fragments, which is consistent with the increase in the top core layer. Not much change was recorded for organic content after LOI analysis at 550 °C as well as for carbonic content at 950 °C; however, a significant amount of carbonic content increase was recorded from 2003 to 2008 (Fig. 4).

### *3.2. Kocaçay Delta*

Due to high mixing and sedimentation rates (7.74 kg m$^{-2}$ y$^{-1}$ on average) at the sampling point of Kocacay Delta, the core could only be dated back to 2015. Although the dating was relatively current, Kocacay core samples had a higher number of microplastics with a higher variety in color and type compared to the Lake Uluabat. The samples were dominated by fibers with a total number of 1297 around 1.7 ± 1.2 mm in size (Table 1). The size of the fibers ranged from 0.18 mm to 4.9 mm. The size distribution along the core was also irregular, as observed in the lake Uluabat. Black fibers (33%) were the most abundant, followed by blue (22%) and red particles (20%) (Figs. 5–6). A high number of fragments with multiple colors were detected in the sample (118 particles per core). Among the fragments, blue (36%) were the most abundant, followed by transparent (17%), white (14%), red (11%) and green (11%) (Fig. 5). Lastly, film particles were also detected in the core with a total of 72 items (Table 2). To identify the chemical composition of microplastics (34 samples) from the Kocacay Delta core, samples were evaluated with FT-IR microscopy. In contrast with samples from Lake Uluabat, only 2 spectra could not be interpreted, while 6 spectra were not conclusive (SI Table 3). 26 microplastics were identified and most of them (22 samples) were polyethylene, while others were polypropylene, polyethylene terephthalate and polystyrene. Polyacrylates and poly (vinyl chloride) microplastics were also identified, but the spectra were not conclusive because of low absorbance and unresolved peaks. We also used 10 additional microplastic samples from the current water surface obtained contemporarily during coring to compare with the polymer composition of microplastics between the core and surrounding environment with FT-IR spectroscopy. Thus, 3 of the samples were identified as polyethylene, while 6 samples were polypropylene and 1 sample could not be interpreted (SI Table 4). Representative selection of FT-IR spectra for the core and the current microplastic samples are also given in SI Figs. 3–5.

## 4. Discussion

To investigate microplastic accumulation, which is a particularly

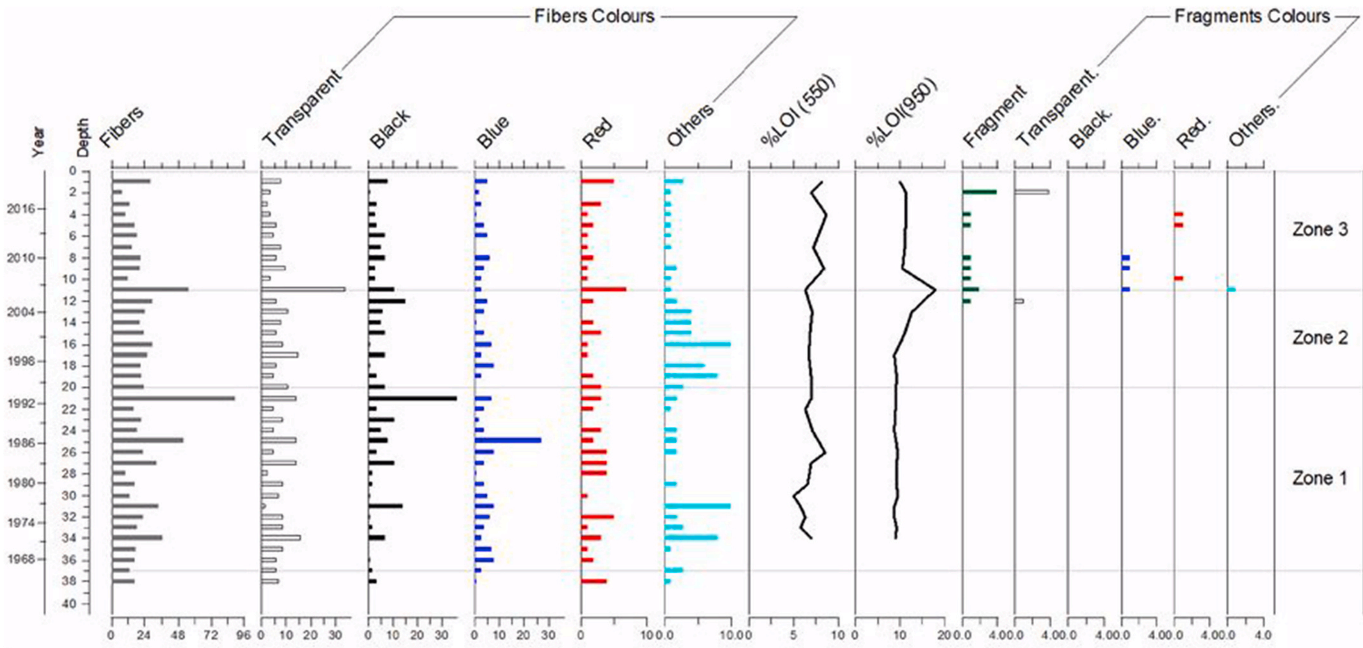


**Fig. 4.** Distribution of microplastic through 1966 to 2020 and LOI values of sediment samples heated at 550 °C and 950 °C in Lake Uluabat.

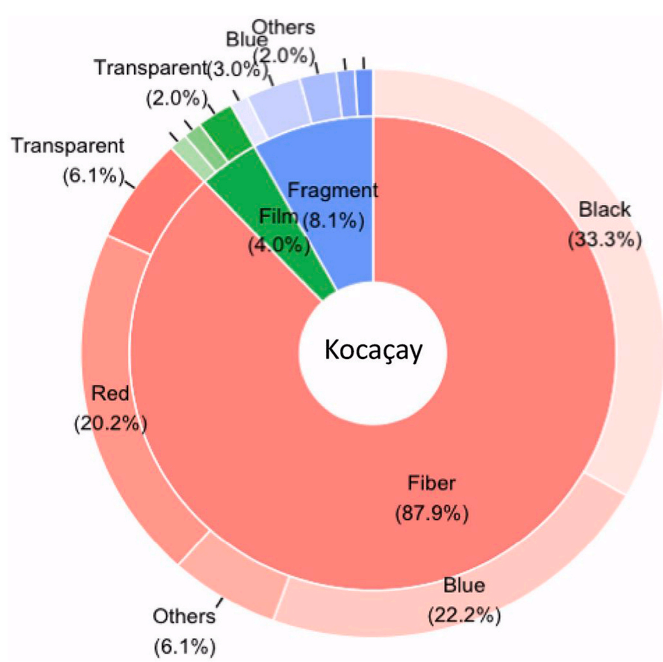


**Fig. 5.** Percent distribution of different colors of observed fibers from Kajak Corer in Kocaçay Delta. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

important proxy for industrial production, paleolimnological perspectives could be a useful tool. Although several studies have investigated microplastic accumulation in sediments of various freshwater ecosystems, to the best of our knowledge there have been only a few studies tracking the microplastic accumulation in lakes over time. One of these studies is the sediment record of microplastics from an urban lake in the UK (Turner et al., 2019). Another study from Lake Ontario, Canada showed microplastic accumulation in a sediment core up to 38 years ago (Corcoran et al., 2015). A study from Donghu Lake in China was dated back 60 years (Dong et al., 2020). Our findings are in line with these two studies, which date back to the 1950s and show that plastic deposits and fibers are abundant microplastic particles. All around the world, plastic pollution is a very important issue; however, monitoring of the microplastic accumulation from the past to the present day has been limited. This limited information is coherent to the freshwaters in Turkey that are subject to several disturbances such as drought, pollution and hydrologic impacts (Coops and BeklioğluCrisman, 2003; Beklioğlu et al., 2020; Coppens et al., 2020).

In Lake Uluabat, from 1966 to the 1990s, an increasing trend of microplastic accumulation was observed. Coincidentally, significant fluctuations in water level were recorded from 1984 to 1996, which can be attributed to low water levels (Levi et al., 2016). During this low water level period, the sediment accumulation rate of the lake may be higher (Kazancı et al., 2004; Reed et al., 2008) and this consequently correlates to high microplastic abundance. Due to high sediment accumulation from the catchment, the lake area decreased from 133.1 $km^2$ in 1984 to 120 $km^2$ in 1993 (İleri et al., 2014). Furthermore, the discharges of waste waters from factories and agricultural runoff influenced the water quality with intensive algal blooms (Magnin and Yarar, 1997; Karacaoğlu et al., 2004; Reed et al., 2008; Yurtseven and Randhir, 2020). Considering the phytoplankton composition and chlorophyll-a concentration before the 1980s, the lake was eutrophic (Torunoğlu et al., 1989). Accordingly, a monthly sampling campaign performed in 1998 and 1999 revealed that the lake had high chlorophyll-a, nitrate, and soluble reactive phosphorus (Dalkiran et al., 2006). Although the physicochemical data were not directly correlated with the microplastic accumulation, these results could give novel information about past pollution in the lake. In 1998, Lake Uluabat and its surrounding area were included in the Ramsar List (Çağırankaya and Meriç, 2013). As a result of this new status at the national level and also by the international community, it has been recognized as having significant value not only for the country but also for humanity as a whole (Ramsar Convention Secretariat, 2010). These measures brought about positive impacts on the pollution status of the lake, which also coincides with the decrease of microplastic numbers recorded in the second zone during the 1996 to 2007 period. However, an increase in microplastics in 2007 coincided with the high income of inorganic/carbonate content at 950 °C. This may be due to higher urban and agricultural waste inputs to the lake causing water pollution (Bulut et al., 2010), and the water level

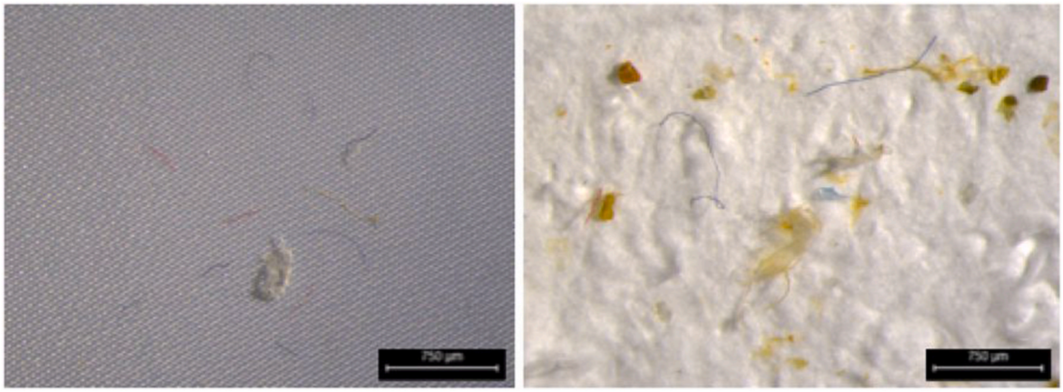

**Fig. 6.** Identified fibers from sediment core samples from Kocacay Delta.

decreased during this period in the lake (Degirminci et al., 2006). There were also several active drainage discharges around the shores of the lake as well as urban and industrial pollution to the lake via the Akçalar stream (Hacısalihoğlu and Karaer, 2020).

From 2008 to 2020, in the third zone, the number of microplastics decreased continuously. The new governing protection policies such as sewage diversion, plastic bag usage, probably triggered a decrease in pollution reaching the lake. However, at the surface sediment layer which represents the year 2020, the recorded microplastic amounts were exceptionally high, compared to the previous years. The lake itself has been actively used for fishing and urban activities for a long time, which probably enhances the mixing of the lake water and sediment throughout the year. During the Covid-19 pandemic lockdown, the mentioned area was possibly less exposed to anthropogenic activities, which might allow microplastic particles to settle onto the uppermost sediment layer without any chance of regular disturbance. The distribution of accumulating sediment is interrelated to lake bathymetry and basin morphology (Hilton et al., 1986), so these factors might control the resuspension and deposition of microplastics in Lake Uluabat. The sediment accumulation rate is more rapid in eutrophic lakes, so the sinking rate of microplastics increases when they are mixed with organic matter (Porter et al., 2018). In addition, the geological and geomorphological structure of the basin of Lake Uluabat, climate, vegetation and the steep slope of the land can cause water erosion in the basin together with anthropogenic factors.

The dominant microplastic particles from the Lake Uluabat core samples were fibers, mainly transparent, black and blue in color. The high amounts of fibers in the samples are in line with reported studies (Willis et al., 2017; Woodall et al., 2015; Horton et al., 2017b; Turner et al., 2019; Li et al., 2020b; Xue et al., 2020). It has been reported that there are 67 residential districts, factories, workplaces, agricultural enterprises and mines in the Mustafa Kemal Paşa stream basin area, which is the main source of the lake (Katip et al., 2015). Hence, dense human population, domestic and industrial wastes and fishing activities are seen as important fiber sources in aquatic systems (Browne et al., 2010; Mintenig et al., 2017; Zhang et al., 2019).

The highly populated Gölyazı village located near the lake lacks agricultural lands, so about 80–85% of the population has been fishing since the 1960s (Degirminci et al., 2006; Aydin and Güngör, 2015). In the 1980s, crawfish catching was popular; however, a parasite infection influenced the population dynamics of crawfish and thereby decreased crawfishing activity after 2000 (Magnin and Yarar, 1997; Çelik, 2000). Thus, the high abundance of fiber could be a result of the degradation of fishing nets and ropes before the 2000s. Currently, this activity has decreased due to environmental pollution, careless fishing habits and migratory fishing birds which have led to a decrease in the economic value of the crawfish. Consequently, the low economic income from fishing in the area is making the local people focus more on tourism as an investment venture (Çelik et al., 2016). Our findings were also coherent with the previous results that fibers were the dominant contaminants. There was no clear distribution pattern for the microplastic abundances over the timescale as observed in a zigzag manner in Lake Donghua (Dong et al., 2020). In contrast to freshwater, limited chronological studies stated that microplastics have displayed an increasing trend at the uppermost layer of deep-sea sediments (Matsuguma et al., 2017; Fan et al., 2019; Chen et al., 2020; Courtene-Jones et al., 2020; Lin et al., 2020). Similar to our results, the sediment core was composed of fibers instead of pellets or debris (Turner et al., 2019; Dong et al., 2020). Similarly, fibers were the dominant type from the deep-sea sediment core records (Matsuguma et al., 2017; Chen et al., 2020; Courtene-Jones et al., 2020; Uddin et al., 2021).

Although the dating of the core from Kocacay Delta was not as clear as the Lake Uluabat core, it can clearly be seen that the high amount of microplastic loading reaching the Marmara Sea originates from the whole Susurluk catchment. Thus, the variety of the microplastics (fibers, fragments, films) found in the sample might be the key factor showing that the discharges of the pollutants from the industrial and urban areas are affecting the whole area to a considerable extent (Dalkiran et al., 2006). Our findings were in line with a study conducted in the Pearl River catchment in China that the variety of microplastics (e.g. fragments, spherules) were higher (Fan et al., 2019). Furthermore, a study conducted on the sediment of the Marmara Sea Shore close to the Kocaçay Delta revealed that the fibers were high (Gürbüz, 2017), which is coherent with our findings in core samples. After the aforementioned study, the score of the microplastic pollution index was found to be moderate in the Marmara Sea Shore close to the Kocaçay Delta (Tan, 2022). A current study from the Golden Horn Estuary of the Marmara Sea stated that smaller size MPs were more abundant than larger ones, and MP accumulation was found in the deep layer of sediment (Belivermiş et al., 2021).

Several factors (e.g. solar radiation, mechanical abrasion, chemical or biological effects) can cause weathering of microplastics. Such processes are initiated at the surface of microplastics and could be followed by a diffusion-controlled process resulting in degraded fractions, i.e. secondary microplastics. Fragmentation of microplastics occurs due to mechanical, photo, thermal and bio-degradation processes or with combinations of them. The degradation process of microplastics also depends on their polymeric nature, mostly chemical composition and age (Li et al., 2018; Veerasingam et al., 2021). Environmental conditions could contribute to the degradation process, and the breakdown of microplastics into smaller fractions complicates the identification of microplastics.

Although paleolimnological perspectives give us an interpretation of past microplastic pollution in freshwater systems, there were shortcomings in the current study during the identification of polymer compositions layer by layer. The main fraction of microplastics consisted of small-sized and thinner-shaped fibers, which limits the methods and instruments used for spectral detection. Raman spectroscopy studies result in destructive effects on the fibers by the laser and therefore this method was not suitable for chemical identification in this study. ATR FT-IR studies result in spectra with low absorbance and unresolved peaks, which corresponds to size limitation for microplastics composition analysis.

Therefore, a representative selection of microplastics on various sediment depths, which were divided into sub-groups by date, was analyzed by FTIR spectroscopy. The spectroscopic evaluation of microplastics revealed that the top layer (zone-3), reflecting the current sediments, has a detectable signal in comparison to the deepest layer in Lake Uluabat. Even though the core sample dated back to the 2000s and reflected the relatively new plastic accumulation in Kocaçay Delta, we detected better spectra from the FT-IR analyses. In conclusion, acquiring better spectra in the top layers could be due to the less weathering and degradation of microplastics in the sediment.

## 5. Conclusions

The dating of sediment records provides extensive knowledge regarding the historical anthropogenic impacts in freshwater environments and the accumulation of such particles in both river and lake ecosystems. In the current study, it was determined that the accumulation of microplastic accumulation dates back to the 1960's in Lake Uluabat, and this is compatible with the beginning of the plastic usage all around the world.

The missing link from past to present on microplastic pollution as an emergent contaminant obtained from paleolimnological perspectives can be revealed for freshwater ecosystems in countries that have no monitoring. Thus, the background information on microplastic pollution would help the implementation of the pollutants by both the researchers and public authorities. However, there is still an urgent need to underline the polymer composition of the microplastics from top to bottom layers. But neither Raman nor ATR FT-IR can detect the clear peak for small size fibers, so more detailed spectroscopic analyses are needed. Since the depletion of microplastic particles are still under debate and many scientific studies have proved that they can be transferred to humans via different microorganisms, it is quite crucial to know the sources of such pollutants and to prevent their influx to the environment again.

## Credit author contribution statement

F.F. Almas: Formal analysis, Investigation, Writing-original draft. G. Bezirci: Conceptualization, Investigation, Methodology, Formal analysis, Visualization, Writing-original draft. A. S. Çağan: Formal analysis, Investigation. K. Gökdağ: Formal analysis. T.Ç.: Formal analysis, Investigation, Writing-reviewing & editing. G. B. Kankılıç: Formal analysis, Investigation, Writing-reviewing & editing. E. Paçal: Formal analysis. Ü.N.Tavşanoğlu: Conceptualization, Methodology, Resources, Supervision, Writing-original draft, Writing-reviewing & editing, Project administration, Funding acquisition.

## 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.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.chemosphere.2022.135007.

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