T.C.
ÇANKIRI KARATEKİN UNIVERSITY
THE INSTITUTE OF HEALTH SCIENCES
THE DEPARTMENT OF ENVIRONMENTAL HEALTH

MICROPLASTIC POLLUTION FROM PAST TO PRESENT IN FRESHWATER ECOSYSTEMS: SUSURLUK BASIN CASE

THE DEGREE OF MASTER OF SCIENCES

Fatma Feisal-ALMAS

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The Institute of Health Sciences
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SUPERVISOR
Prof. Dr. Ülkü Nihan YAZGAN TAVŞANOĞLU

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ACCEPTANCE AND APPROVAL

Fatma Feisal ALMAS, the graduate student of The Institute of Health Sciences with the student number of 198207300, has successfully presented her thesis entitled "Microplastic Pollution from Past to Present in Freshwater Ecosystems: Susurluk Basin Case" before the jury whose signatures are below, after fulfilling all of the requirements determined by the relevant regulations for the degree of Master of Science:

Date:

Chairman:	**Prof. Dr. Ülkü Nihan YAZGAN TAVŞANOĞLU** **Çankırı Karatekin University**	………………………… **Signature**
Member:	**Assist. Prof. Dr. Okan ÜRKER** **Çankırı Karatekin University**	………………………… **Signature**
Member:	**Assist. Prof. Dr. Tamer ÇIRAK** **Aksaray University**	………………………… **Signature**

Approved for the Graduate School of Health Sciences

Prof. Dr. Özcan Özkan
Director of Graduate School

ETHICS STATEMENT

The thesis entitled “Microplastic Pollution from Past to Present in Freshwater Ecosystems: Susurluk Basin Case” which was prepared and presented as a thesis, was written by myself and in accordance with the scientific, academic rules and ethical conduct. The idea/hypothesis of my thesis solely belongs to my supervisor and to me. The research pertaining to the thesis was conducted by myself and therefore, all of the used sentences and interpretations within the work belongs to me.

I declare the aforementioned issues to be correct.

Signature
Date
Fatma Feisal Almas

PREFACE AND ACKNOWLEDGEMENTS

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CONTENT

INDEX OF ABBREVIATIONS AND SYMBOLS

C : Carbon

Ca : Circa

CRS : Constant Rate of Supply

Cs : Caesium

E : East

H_2O_2 : Hydrogen Peroxide

LOI : Loss on Ignition

N : North

Pb : Lead

Ra : Radium

PAH : Polycyclic Aromatic Hydrocarbons

PCB : Polychlorinated Biphenyls

POP : Persistent Organic Pollutants

UK : United Kingdom

^{o} : Degree

^0C : Degrees Celsius

Cm : Centimetre

keV : Kilo-electron Volts

Kg : Kilogram

km^2 : Kilometre squared

m : Metre

ml : Millileter

mm : Millimetre

µm : Micrometre

% : Percentage

X : Times

Y : Year

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TATLISU EKOSİSTEMLERİNDE GEÇMİŞTEN GÜNÜMÜZE MİKROPLASTİK KİRLİLİĞİ: SUSURLUK HAVZASI ÖRNEĞİ

ÖZET

ALMAS, Fatma Feisal. Tatlısu Ekosistemlerinde Geçmişten Günümüze Mikroplastik Kirliliği: Susurluk Havzası Örneği, Yüksek Lisans Tezi, Çankırı, 2021.

Küresel ölçekte değerlendirildiğinde sucul ekosistemlerde mikroplastik kirliliği son yıllarda önemli bir sorun haline gelmiştir. Bu kirliliğe yönelik çalışmaların çoğunluğu denizel ortamlarda gercekleştirilmiştir ancak taşınım yolları dikkate alındığında tatlı su ekosistemlerindeki plastik kirliliğinin araştırılması önemli bir konu haline gelmiştir. Dünyanın pek çok bölgesinde sanayi devrimiyle birlikte artan plastik kullanımının sucul ekosistemlerdeki birikimine ilişkin tarihsel veriler sınırılıdır. Bu çalışmada, insan faaliyetlerinin ve buna bağlı kirletici yükünün yüksek olduğu Bursa bölgesinde mikroplastik kirliliğinin zaman içerisindeki birikimi, Uluabat Gölü ve Kocaçay Deltası'nda paleolimnolojik yaklaşımlardan faydalanılarak incelenmiştir. Her iki örnekleme noktasının dip sedimanından iki kısa karot örneği alınmış ve bu sedimanlar hem yaşlandırma hem de profil boyunca biriken mikroplastiklerin belirlenmesi için kullanılmıştır. Mikroplastik kirliliği, Uluabat Gölü'nde 1960'lı yıllardan itibaren başladığı tespit edilmiştir. Özellikle fiber baskınlığının yüksek olduğu gölde bu birikimin balıkçılık faaliyetlerinin yoğun yapılmasıyla ilişkili olduğu düşünülmektedir. Kocaçay Deltası'nda ise sedimanın sürekli karışımı nedeniyle göl sistemindeki kadar iyi bir yaşlandırma mümkün olamasa da 2014 yılından itibaren birçok farklı tipte mikroplastiğin birikimi tespit edilmiştir. Ülkemizde ilk kez bir tatlı su ekosisteminde mikroplastik kirliliğinin zamansal profilinin çıkarıldığı bu çalışma; mikroplastiklerin paleolimnolojik yaklaşımlarda iyi bir belirteç olarak kullanılabileceğini göstermiş ve bu şekilde hem geçmisteki plastik birikim profilinin anlaşılması hemde geleceğe yönelik alınabilecek koruma yöntemleri ile ilgili veri sağlanması açısından önemli bir araç olduğu ortaya konumuştur.

Anahtar Kelimeler: Kirlilik, Mikroplastik, Paleolimnoloji, Tatlısu Gölleri.

MICROPLASTIC POLLUTION FROM PAST TO PRESENT IN FRESHWATER ECOSYSTEMS: SUSURLUK BASIN CASE

SUMMARY

ALMAS, Fatma Feisal. microplastic pollution from past to present in freshwater ecosystems: susurluk basin case, Master Thesis, Çankır, 2021.

When evaluated on a global scale, microplastic pollution in aquatic ecosystems has become an important problem in recent years. Most of the studies on this pollution have been carried out in marine environments, but considering the transport routes, the investigation of plastic pollution in freshwater ecosystems has become an important issue. Historical data on the accumulation of plastic use in aquatic ecosystems, which increased with the industrial revolution in many parts of the world, is limited. In this study, the accumulation of microplastic pollution over time in Bursa region, where human activities and related pollutant production is high, was investigated by using paleolimnological approaches in two selected points, Uluabat Lake and Kocaçay Delta. Two short core samples were taken from the bottom sediments of both sampling points and these sediments were used for both dating and determination of microplastics deposited from top to bottom. It has been determined that there is Microplastic accumulation in Uluabat Lake starting from the 1960s. Especially in the lake where fiber dominance is high, this accumulation is thought to be related to the intense fishing activities. In the samples analyzed in the Kocaçay Delta, many different types of microplastics have been accumulated since 2014, although its dating was not as good as in the lake system due to the continuous mixing of the sediment in the Delta. In this study, in which the temporal profile of microplastic pollution in a freshwater ecosystem is researched for the first time in our country, it has shown that microplastics can be used as a good indicator in paleolimnological approaches, and in this way, it has revealed that an important development can be achieved both in understanding the plastic accumulation profile in the past and in providing data about the protection methods that can be taken for the future.

Key Words: Freshwater Lakes, Microplastics, Paleolimnology, Pollution

1. INTRODUCTION

1.1 Freshwaters

Freshwaters are vital in the world since they serve varied habitats for various organisms and function as a transition zone between terrestrial and aquatic ecosystems; hence they help in sustaining complex ecological communities (Moss, 1998; Beklioğlu et al., 2011). Freshwaters cover 0,01% of the water on Earth and only 2,3% of the surface area comprises lakes, rivers, wetlands and resorvoirs exculuding ice sheets (Lehner and Döll, 2004). At the same time, freshwaters are the most diverse ecosystems and harbour more than 14 0000 species (Meng et. al., 2020). Freshwaters play an important role in maintaining regional and hydro-climatic regimes serving several ecosystem services through natural filtration and maintaining the water quality (Jeppesen et al., 1997; Scheffer, 1998; Søndergaard and Jeppesen, 2007). Moreover, they are invaluable to humans for providing drinking water, food and energy and have a crucial role in transportation, waste disposal and remediation, control erosion and flooding, industrial and recreational activities (Moss, 1998; Meng et. al., 2020), such as fishing, kayaking, river rafting, etc. (Bruce Aylward et al., 2005). The majority of the world's freshwater ecosystems are made of small shallow lakes (Moss, 1998; Wetzel, 2001). A lake with less than 3 m mean depth with an unstable thermal stratification brought about by a continuous mix up in its water column is categorised as shallow lake (Moss, 1998). Freshwater shallow lakes and rivers are simultaneously characterised by the presence of macrophytes in their wide littoral area which survive on the light that penetrates through the shallow waters (Moss, 1998; Beklioglu et al., 2011) therefore they have a richer community of different species than in deeper lakes (Jeppesen et al., 1997). However, because of their close system structure, they tend to be more prone to nutrient re-suspension caused by disruptions from climate change and human activities than deeper lakes (Padisák and Reynolds, 2003; Beklioglu et al., 2011; Gottschalk and Kahlert, 2012; Dokulil, 2014). Different freshwater bodies face different types of pollution depending on their region. Pollutants from factory discharges, agricultural run-offs (fertilizers, pesticides, livestock waste, sewage and pharmaceuticals), macro- and microplastics may have direct or indirect and sub-lethal or lethal effects with unforeseen outcomes (Dudgeon, 2019). Excess anthropogenic disturbances continue to endanger the freshwater species and ecosystems (Dudgeon et al., 2006). Also, these anthropogenic disturbances are known to cause hydrological modifications, habitat degradation, water pollution and invasion, river diversions, climate change, rising sea levels and over exploitation of freshwater bodies (Lin et al., 2017; Albert et al., 2020). In the presented study we focused on microplastic pollution on a freshwater lake.

1.2 Microplastic Pollution

Currently at 368 million metric tonnes of plastic production recorded (Statista, 2021) and the production has been continuously increasing since it was first produced as a synthetic polymer *Bakelite*in 1907. Thus, several authors stated as Plastic Age in the current period (Cózar et al., 2014; Thompson et al., 2004). Due to their utilizable characteristics which are high durability, flexibility, ease of processing, lightness, reusability and most of all affordability would trigger the increase in using plastic in almost all sectors (Cózar et al., 2014; 52 Patchaiyappan et al., 2020, Bertoldi et al., 2021). Unfortunately, unadaquate waste management of the plastics reach the aquatic environment (de Sá et al., 2018; Du et al., 2021). Microplastics are defined as plastic debris smaller than 5 mm in size (Fendall and Sewell, 2009; Hidalgo-Ruz et al., 2012) and generated by antropogenic activities (Horton et al., 2017) which originated from primary and secondary sources. The primary microplastics are industrially produced for different uses according to their sizes. Microbeads are industrially produced and used in personal care products and cosmetics, as exfoliates and cleaning agents, abrasives, textile microfibers and pellets are used as pre-production pellets (Rochman et al., 2019). Secondary microplastics are those that come about from breakage and degradation, from larger plastic waste (plastic bags, fishing nets, single use plastics, etc.) via natural systems like photo degradation, errand physical scuffing which bring about thinning of the plastic surfaces hence increasing their fragility (Andrady, 2011; Zettler et al., 2013; Portolés Pérez, 2020) (figure 1.1).

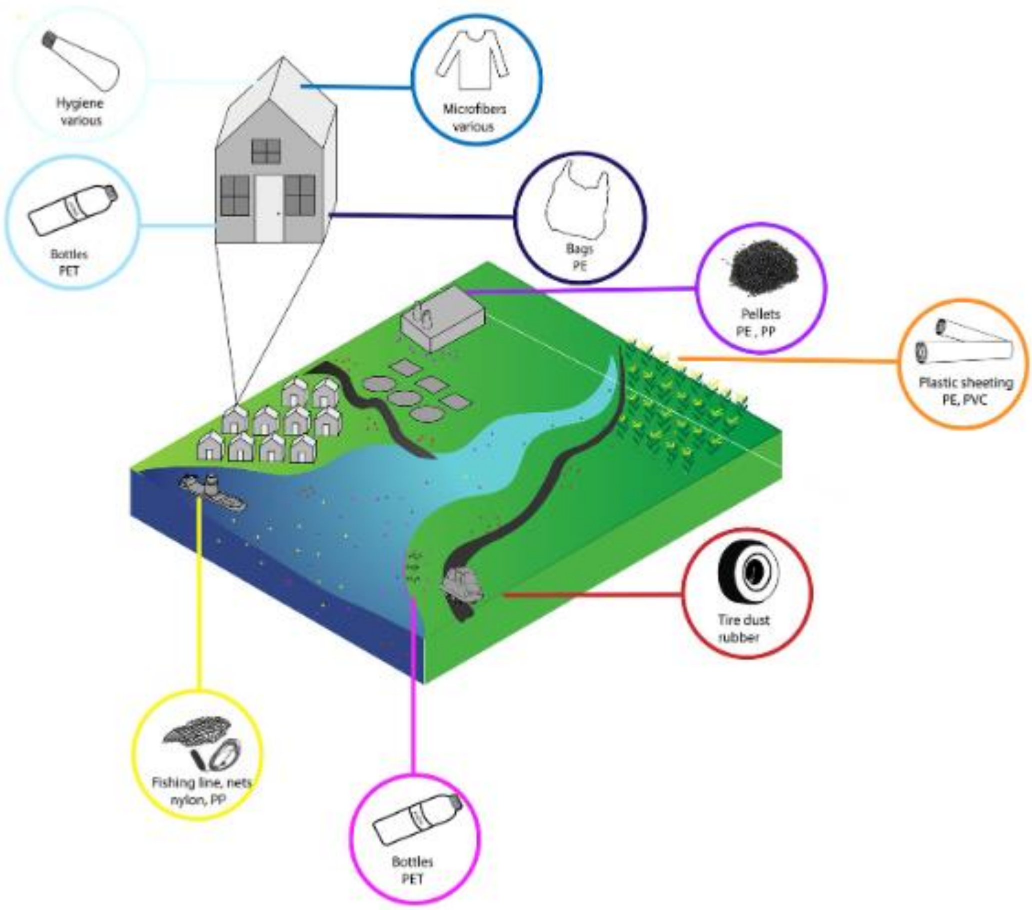

Figure 1.1: Different types of primary and secondary microplastics (Rochman et al., 2019).

Both primary and secondary microplastics bring about different types of microplastics in the aquatic ecosystems such as fragments, pellets, fibres, films and foams (Figure 1.2).

Figure 1.2: Different types of microplastics (Photo: Dr. Gizem Bezirci).

Different types of microplastics are transported throughout the ecosystem via several ways such as surface runoffs, atmospheric deposition, sewage treatment plants and soil erosion (Browne, 2015; Horton et al., 2017; Akdogan and Guven 2019; Amrutha and Warrier, 2020; Hitchcock, 2020;). It is estimated that the plastic waste discharged into the ocean every year through freshwaters especially rivers and lakes during extreme floods reaches approximately 4,8 to 12,7 million tonnes (Bläsing and Amelung, 2018; Gündoğdu et al., 2018; Bertoldi et al., 2021). Plastics are organic and/or non-organic compounds-based materials mostly made of polymers obtained from non-renewable crude oil (Lithner et al., 2011). Since, mostlynthey are practically indestructible, it is making them difficult to decompose by organisms exposed to them. In addition, when microplastics sink into the sediments and bioaccumulate there, the amount is bound to increase through biofouling.

Consequently, plastic particles bioaccumulate in the bodies of the exposed organisms and tend to cause harmful health effects (Miller Id et al., 2020). Also, the surfaces of microplastic are a suitable habitat for microbial colonisation creating biofilms with pathogenic microorganisms which bring an increase in the toxic potentiality of microplastics in aquatic ecosystem (Tavşanoğlu et al., 2020) since a rich microbial biofilm has been observed to increase microplastic actractiveness as food for several marine organisms (Esensoy et al., 2020). Furthermore, microplastics are a sink for toxic chemicals like POPs, PCBs, PAHs, trace metals and organochlorine pesticides. These toxic chemicals attach to microplastic surfaces and affect aquatic organisms exposed to them as well (Rios et al., 2007; Ren et al., 2020). Microplastic pollution is a relatively new topic; even so it is considered an emerging concern contaminant for the whole environment (Lambert and Wagner, 2018). Because of their high persistency and significant accumulation rates in different environments, it is crucial to evaluate the occurrence and types of microplastics through time. According to their deposition contexts, lake ecosystems provide an archive representing catchment scale sinks for microplastic debris. A sediment core from Santa Barbara Basin, California was observed to have a concentration of fibres in the top 15 cm of the core dating to over 600 years (Brandon et al., 2019). A sediment core from a North London Lake observed microplastic upto 539 particles per kilogram of dried sediment datin from the late 1950s (Turner et al., 2019). In another sediment profile collected from Kuwait Bay also observed a total of 76 microplastics, 52 of them being fibres (Uddin et al., 2021). Generally, microplastics contamination are observed to prevail in bottom waters than higher water surfaces which eventually get deposited and buried into sediment profiles (Peng et al., 2018).

1.3 Effects of Microplastic on Freshwater Biota and Ecosystems

Research on the effects of microplastic pollution on marine organisms shows organisms to be affected with internal injuries even blockage of intestines, malnutrition and death due to excessive feeding of microplastic in daily natural diet of those organisms (Derraik, 2002; Lusher et al., 2015); however, research on freshwater biota related with this prior subject still has gaps (Scherer et. al., 2018). In freshwater food web, organisms have different feeding behaviour such as predators, scrapers (grazers), schreders, suspension feeders, deposit feeders, filter feeders, collectors (Scherer et. al., 2018).

Microplastics as being in the same range size of particles ingested as food for these organisms, these particles can easily get mixed up with the actual food source and hence can be easily consumed (Galloway et al., 2017). Larger plastic debris has also been reported to have fatal effects on organisms ingested by sea turtles and problematic on entangled seabirds (Meng et. al., 2020).

Filter feeders such as *Daphnia magna* that feed on the microplastic contaminated muddy surfaces, have shown adverse effects observed in laboratory experiments (Scherer et. al., 2018). Zooplanktons are mostly exposed to microplastics highly present in surface waters where they acquire their food (Cózar et al., 2014). However, microplastic ingestion varies according to the taxa, life-stage, microplastic size and also external carapace and appendages of exposed zooplankton (Cole et al., 2013). Consequently, zooplankton play a major role in the direct uptake of microplastic into the food web at low trophic levels and from that point, microplastic particules can indirectly travel up to upper-level organisms through prey predator interactions (Mato et al., 2001; Teuten et al., 2009; Cole et al. 2011; Rochman et al. 2014; Scherer et. al., 2018; Aytan et al., 2022).

1.4 Palaeolimnology

Throughout earth’s history, lakes have formed and dried out, however their traces have been preserved in their shorelines, sediments and sedimentary rocks. Sediments that are relatively undisturbed, acts as a valuable stratigraphic archive for their lake environments (Smol, 2008). The constructive sediments preserve diverse information from past status of both aquatic and terrestrial communities (Wohlfarth B., 2012). Paleolimnology is a multidisciplinary field that uses the physical, chemical and biological information (i.e., proxy data or indicators) from both allochthonous (outside the lake) and autochthonous (within the lake) (Figure 1.3) to reconstruct environmental, hydrological and climatic changes of the lakes and their response to the changes through time (Frey, 1988; Smol et al., 2005).

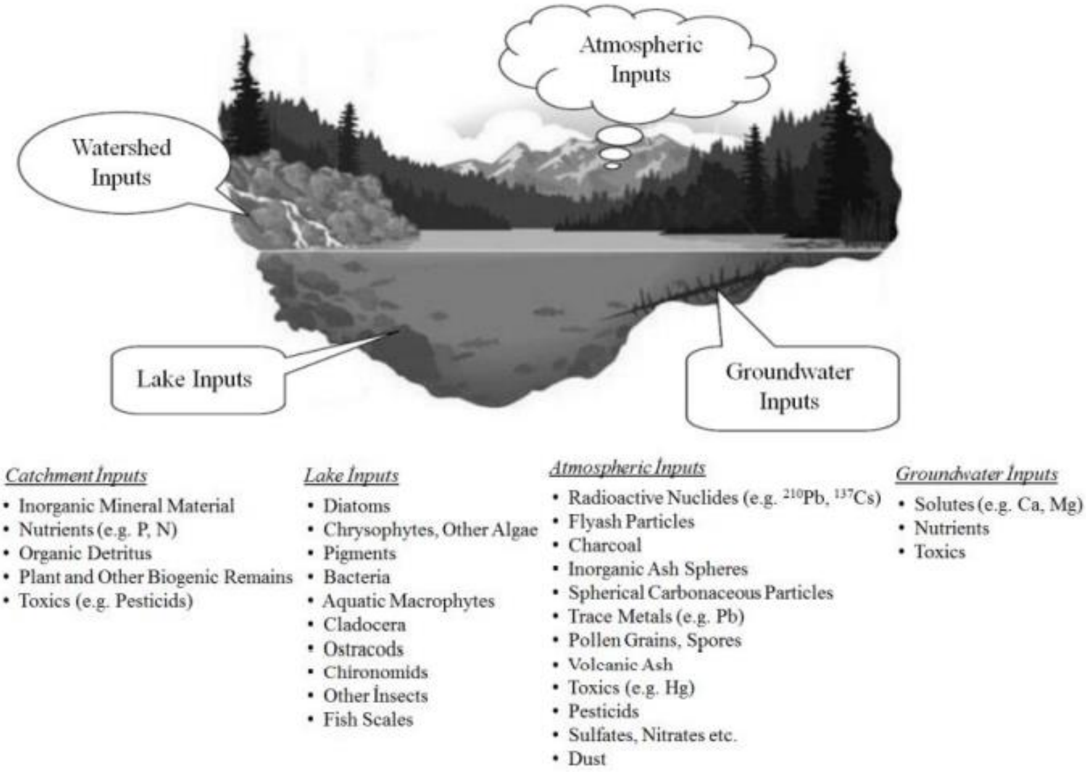

Figure 1.3: Examples of allochthonous and autochthonous inputs into the lake sediments (Smoll, 2008; Eti. E. Levi, 2016).

The first palaeolimnological studies begun in the early 1900s and has since been improving evidently (Douglas, 2013). Since then, developed approaches have been used in an effort to reconstruct previous environmental status of aquatic systems such as the transfer function approach which was the interpretation of past conditions of freshwater ecosystems (Birks, 1998; Hall and Smol, 1999; Last and Smol, 2001). Currently, both single and multi-proxy studies are being used in reconstructing the past environments and climate change (Birks and Birks, 2006; Lotter, 2003; Douglas, 2013). The information from palaeolimnology can be used to management of the lakes (Smol, 2008; Battarbee, 1999; Last and Smol, 2001) as well as understanding the changes through time.

Information on paleoenvironmental conditions such as paleoclimate and paleo productivity can be retrieved from physical sediment characteristics (e.g., grain size) and geochemical characteristics (e.g., isotopes) (Frey, 1964; Douglas, 2013). Organic and carbonate contents of the sediments can be obtained by Loss on Ignition process (LOI) while the industrialisation process around the lake can be traced by physical proxies such as charcoal and fly ash particles (Heiri, Lotter, and Lemcke, 2001; Douglas 2013).

Sediment corers such as kajak corers, gravity corers and piston coring (figure 1.4) are used to extract sediment samples straight down from lake bottoms. According to the "Law of Superposition" which is the main principle of paleolimnological studies, if the sediment is not disturbed during its formation sequence, its deepest layer represents the oldest layer and its age will decrease towards the surface to the topmost layer being the youngest (Douglas, 2013). Thus, the retrieved core will contain an accumulation of a depth-time profile (Smol, 2012) which can be dated using the likes of radioactive decay dating techniques (e.g., ^{210}Pb and ^{14}C) (Smol, 2012; Douglas, 2013).

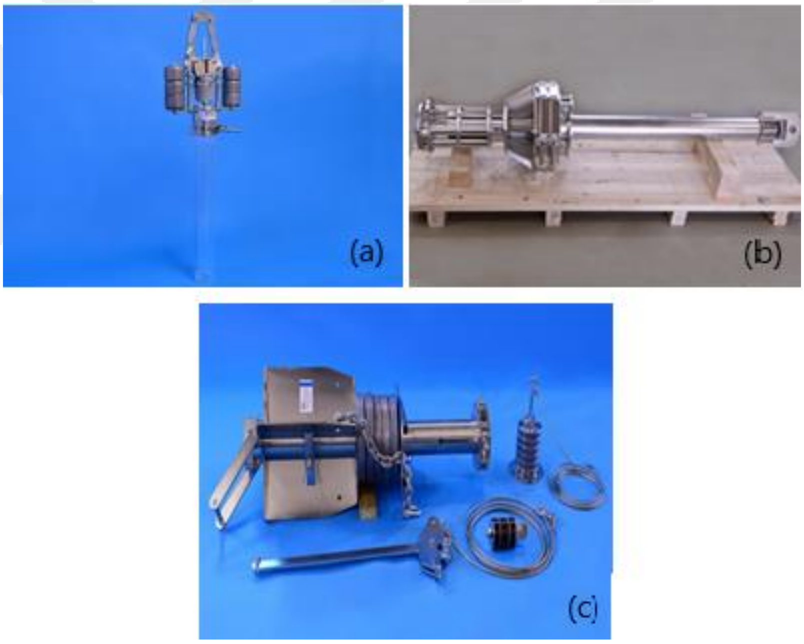

Figure 1.4: a) Kajak Corer, (B) Gravity Corer and (c) Piston Corer (https://www.kc-denmark.dk/products/sediment-samplers.aspx).

1.5 Microplastics as a New Proxy in Palaeolimnology

Compared to marine ecosystems, research on plastic accumulation in freshwater ecosystems is quite new (Wagner et al., 2014; Horton et al., 2017; J. Li et al., 2018). However, plastics are extremely persistent and they accumulate in different compartments in the ecosystems at a high rate, so it is important to evaluate the occurrence, type, and size through time (Uddin et al., 2021). According to deposition contexts, lake ecosystems provide valuable information as catchment scale sinks for microplastic debris. In Turkey, there are some paleolimnological studies using biotic proxies such as subfossil cladoceran, diatoms, macrophytes, polens etc. to evaluate the past environmental changes (e.g. Çakıroğlu et al., 2014; Bezirci, 2017; Reed et al., 2008; Ülgen et al., 2012; Levi et al., 2016). Although, several studies have reported on microplastics in surface waters, but there are relatively few researches on plastic accumulation through time in freshwater habitats compared to marine ecosystems. According to Web of Science (WOS), there are 4063 papers published related to microplastics from 2016 to 2021. Among these papers, 553 of them are related to microplastic presence on freshwater ecosystems. However, microplastics in sediment layers have not been actively used to reveal their annual deposition. From 2016 to 2021, WOS results showed that only 6 papers were published related to microplastics deposion on sediment records. Among the few and current studies on microplastic deposition in sediment records, a study from an urban lake in UK (Turner et al., 2019) observed microplastic in core sediments which dates back to the late 1950's. Another study from Lake Donghu, Wuhan City in China was dated back to 1963 (Dong et al., 2020). From Lake Ontario, Canada, five sediment core samples collected and a sedimentation rate of 1,77 mm/year were tested (Ballent et al., 2016). Another study, also reported microplastic observed from sediment core samples collected form a canal in Tokyo Bay and Sakurada-bori Moat in Japan, Gulf of Thailand, straits of Johor in Malaysia and Durban Bay in South Africa (Matsuguma et al., 2017). A study from the Golden Horn Estuary, Sea of Marmara in Turkey observed a microplastic present in sediment core dating to early 1900s (Belivermiş et al., 2021). In this context, microplastics would be used as a new proxy in palaeolimnology for tracking microplastic accumulation rates in freshwater lake from ca. 150 years period. Due to the absence of long-term records on microplastic pollution, historical information can prove invaluable for both researchers and authorities to help in forecast future trends of plastic pollution in a conservation perspective. Investigation of environmental archives that trap microplastic and accumulate them, help in evaluation of their occurrence, usage patterns and different types of plastic through time (Turner et al., 2019). Paleolimnological approach enables us to establish the Anthropocene period with microplastic as a potential indicator of this period and the effects brought about to the status of freshwaters (Dong et al., 2020; Çevik et al., 2021).

1.6. Research Aims and Objectives

The aim of this study was to investigation of microplastic pollution and distribution over time in a Ramsar site Lake Uluabat, and Kocacay Delta that connects to Marmara Sea directly, with the intention of assessing the effects of anthropological activities onto the freshwater ecosystems.

The detailed research objectives are as follows:

I. To measure the amount of microplastic accumulation over time in two dated sediment cores.

II. Research on the correlation of anthropological activities with the types of microplastic accumulated around the area.

2. MATERIAL METHODS

2.1. Study Site

In order to investigate the microplastic accumulation rate in a Susurluk river basin, two sites were selected from Bursa province of Turkey (Figure 2.1). Bursa (40.1885º N, 29.0610º E) is a hub for industrial and agricultural manufacture with many different sectors such as textile, dye industry, drug and cosmetic manufacture, automotive and agriculturally based production which is making it the fourth most populated city in Turkey (Teksoy et al. 2017). The excessive anthropogenic activities that take place in the areas around the sampling sites are certainly among the main factors contributing to plastic pollution in LakeUluabat and Kocacay Delta (Iscen et al., 2008).

The first sampling site was Kocacay Delta which is 21 km in length and 3,5 km at its widest (Irtem and Sacin, 2012) connects with Marmara Sea from the south (Figure 2.2). Fishing and farming are a popular income for inhabitans around the river. The major water sources of the Kocacay River are Susurluk River and Nilufer Stream which are highly polluted (Tavşanoğlu and Akbulut, 2020).

The second sampling site was Lake Uluabat (40.1489º N, 28.6148º E) (Figure 2.3) with a mean annual depth of 2,5-3 m and, covers up an area of about 155 km^2 during the highest water level periods and 135 km^2 at lowest water level state (Dalkıran et al., 2006). The main influx of Mustafa Kemal Paşa Stream where the polluted water from industrial discharge, sewage effluents, agricultural waste and waste from mining activities flow into the lake (Değirmenciet al., 2006). The lake serves as an important freshwater source for the region and was included on the list of RAMSAR sites in 1998 due to its rich biodiversity with relation to aquatic plants and it is a prime location for migratory bird route (Salihoglu and Karaer, 2004).

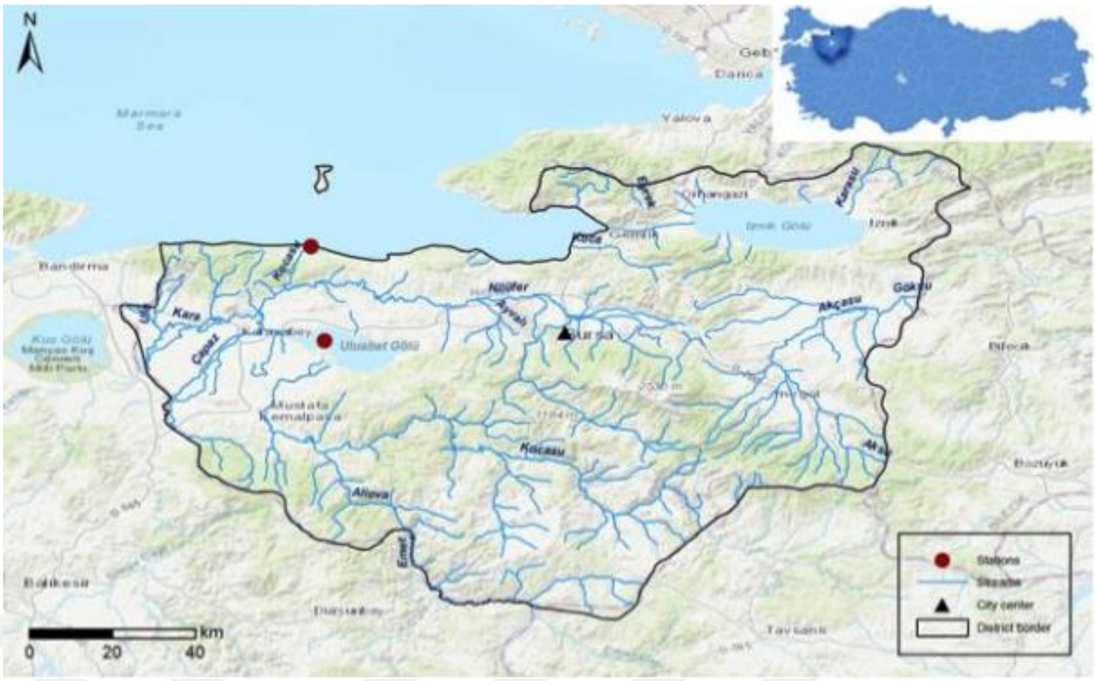

Figure 2.1: The map shows the general view of Bursa region and the red dots points out the sampling sites for the study.

Figure 2.2: Kocaçay Delta (Photo: Ü. Nihan Tavşanoğlu, 2020).

Figure 2.1: Lake Uluabat (Photo: Ü. Nihan Tavşanoğlu, 2020).

2.2. Paleolimnological Sampling and Dating

Accoding to a bathymetric map by Kazancı et al., 2004 and Aksoy et al., 2016, two cores per each station were taken from the deepest points of Lake Uluabat and Kocacay Delta (Figure 2.4). According to the "Law of Superposition" the deepest layer represents the oldest layer and its age will decrease towards the surface, to the topmost layer being the youngest (Douglas, 2013). One core was for dating and the other one was used for investigating the microplastic abundances.

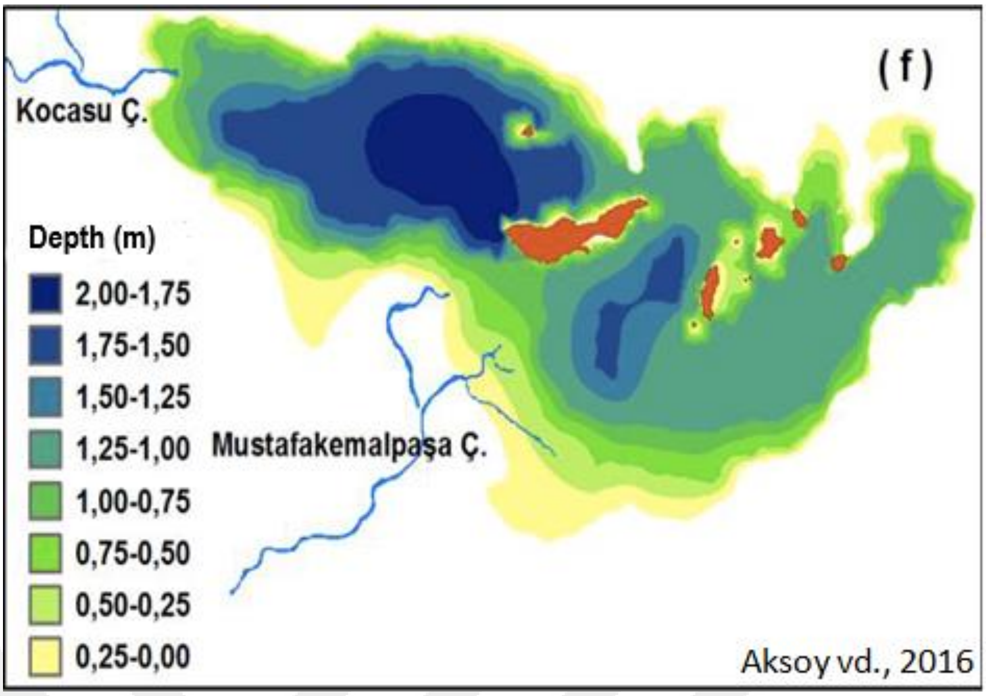

Figure 2.4: Bathymethry map of Lake Uluabat taken by Aksoy et al., 2016.

Sediment samples were extruded by using a KC-Denmark Kajak Corer (32/20 short corer) (Figure 2.5). A pair of 38 cm and 17 cm core samples was obtained separately for Lake Uluabat and Kocacay Delta, respectively. Using a stainless-steel blade in the field, the cores were sliced into 1 cm thick subsamples (Figure 2.6) and stored at +4°C until laboratory analysis. After every slice, the tools were thoroughly rinsed with distilled water before the next slice was extruded.

Figure 2.5: Core sampling (Photo: Ü. Nihan Tavşanoğlu, 2020).

Figure 2.6: Core slicing (Photo: Ü. Nihan Tavşanoğlu, 2020).

One batch of the subsamples was sent for dating and the other batch was used for the laboratory analysis. For each site, sediment core was dated at the Gamma Dating Centre, Institute of Geography, University of Copenhagen. The chronology of the cores was calculated using the constant rate of supply (CRS)-modelling using a modified method (Appleby, 2001; Andersen, 2017). Sediments from both sites were analysed 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, 5keV, ^{226}Ra via the granddaughter ^{214}Pb (peaks at 295 and 352 keV) and ^{137}Cs via its peak at 661 keV. Loss on ignition (LOI) analysis at 550°C and 950°C was also performed for each layer (Heiri et al., 2001).

2.3. Laboratory Analysis

The sliced sediment samples were sieved through a 100 μm stainless steel filter while washing with distilled water until most of the sediment had been removed (Figure 2.7). If the sediment was not removed due to the grain size of the mud, we performed density separation via NaCl. Plastics have a low density thus they tend to be buoyant and float in NaCl solution while denser inorganic clay from the sediment sinks (Li et al., 2018). To retrieve denser microplastics that may have still be in the sediment deposits, organic separation was applied. The deposit was then put in a glass jar and was treated with Fenton's reagent (a mixture of 30% H_2O_2 and $FeSO_4.7H_2O$) at 45 °C on the hot plate as shown in (Figure 2.8). This allows for organic matter present in the sediment to be digested which makes it easier during visual identification of microplastics (Prata et al., 2019).

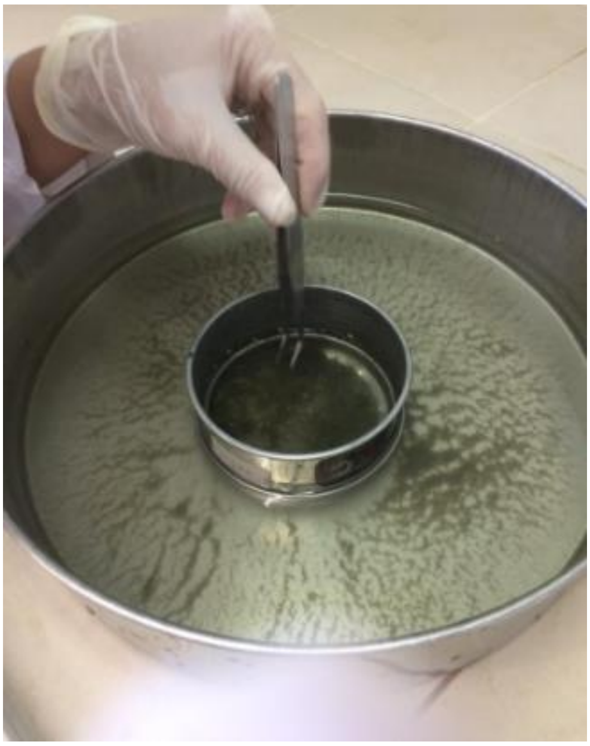

Figure 2.7: Sediment Filtration (Photo: Elif Paçal, 2020).

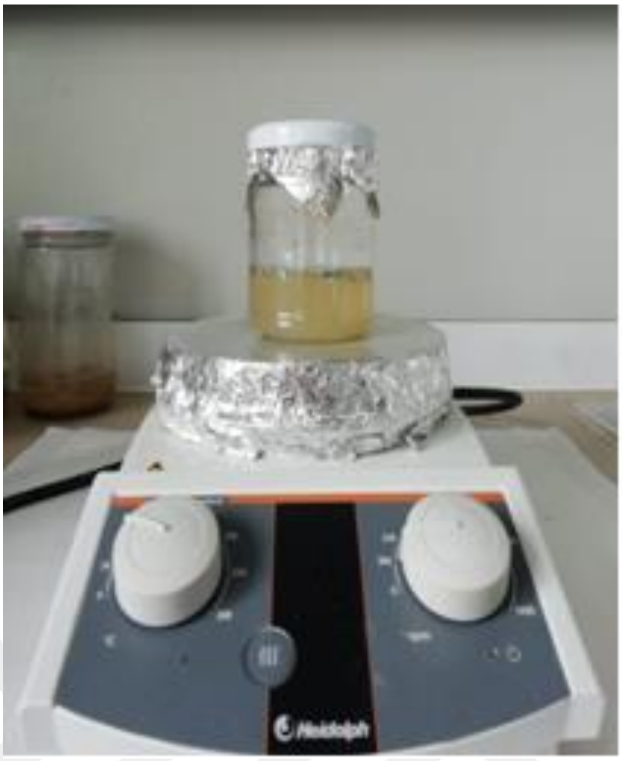

Figure 2.8: Organic removal from sediment. (Photo: Gökben Başaran Kankılıç, 2020)

The samples were not chemically treated for more than 5 days. Afterwards, the samples were filtered through a GF/C Whatman filters with a pore size of 0,45 μm under stainless steel vacuum filter. Before filtration begun, the stainless-steel vacuum filter was washed using distilled water and it was placed under cover using foil to prevent airborne contamination until usage. The residue on filter papers were placed to clean glass petri dishes (Figure 2.9) and kept in the oven for 24 hours at 45 °C to prepare dry samples for microscopic analysis (Figure 2.10).

Figure 2.9: Samples arranged in the oven for drying (photo: Melike Seyfe, 2020).

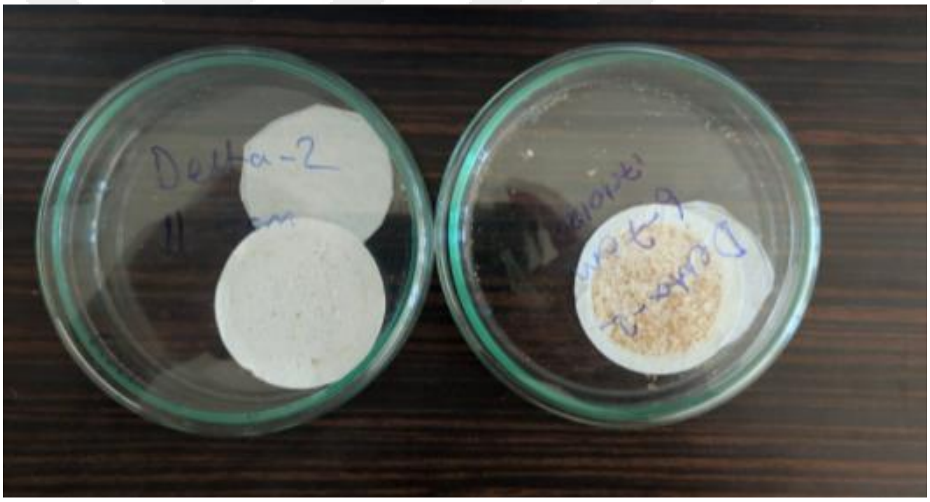

Figure 2.10: Sediment sample after process (Photo: Ali Serhan Çağan, 2021).

For microplastic identification, Leica MZ 80 Flexicam C1 attached Stereomicroscope were used with magnification of 1.75X to 32X. The microplastic wereclassifiedaccording to their type and colour by using tweezers pens and needles (Hidalgo-Ruz et al., 2012). Fibres were examined for equal thickness throughout their length, those that were observed to have branches like organic hairs and plant fibres were not recorded down as fibres and films were critically examined to ensure they did not have any cellular organs visibly present (Turner et al., 2019). Subsequently, identified microplastic samples were classified into different sizes under the stereo microscope.

Throughout the laboratory procedures, cotton laboratory coats were worn and any unnecessary contact with plastic equipment was avoided.

2.4. Data analysis

Each microplastic sample was measured to assessthe sizes using open-source scientific image-analysis program, ImageJ (Abramoff et al., 2004). C2 program was used to analyse core data and the production of stratigraphic diagram of microplastic distribution along the core samples (Juggins, 2007).

3. RESULTS

3.1. Microplastic Distribution and Abundance

Sediment samples from Kocacay Delta contained different colours of fibres, fragments and films (Figure 3.1). Fibres were the dominant observed microplastic type in a total number of 1297 with sizes varying from 0,4 mm to 4,9 mm. (Table 1). Black fibres were observed to be the most abundant (38%) succeeded by blue (26%) and red particles (24%). Fragments with various colours were observed with a total of 118 particles in core. A total of 72 films with different colours were also recorded (Table 1).

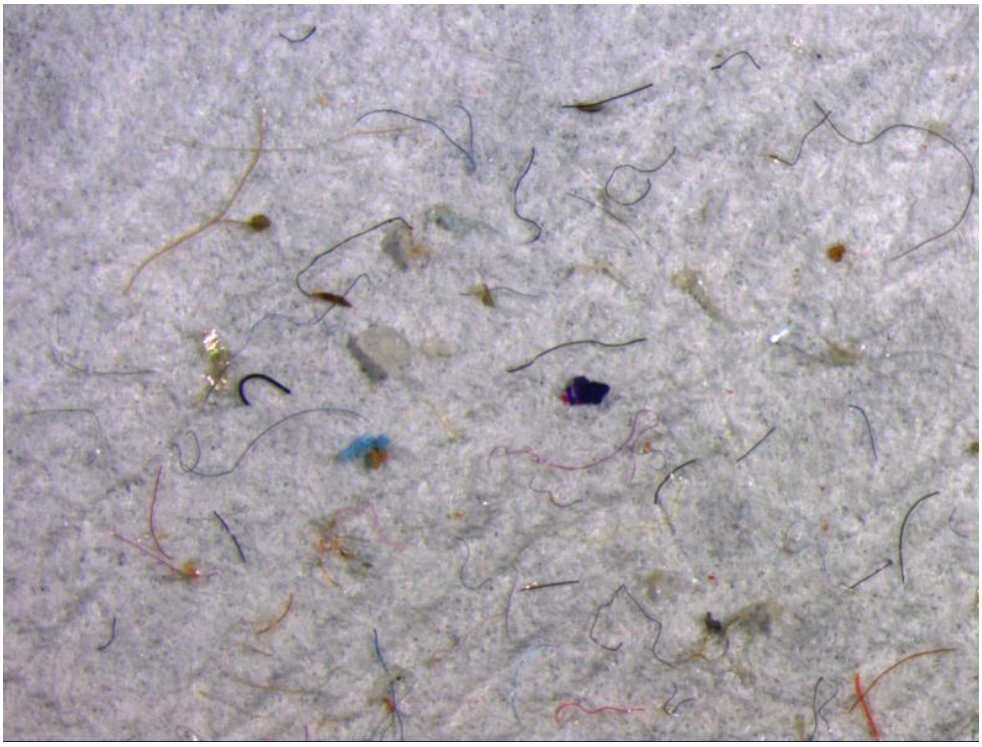

Figure 3.1: Different types of microplastics retrieved from Delta sediment layer (Photo: Ali Serhan Çağan, 2021).

Table 1:Distribution of microplastics in Kocacay 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

Sediment samples from Lake Uluabat were observed to contain mainly fibres (Figure 3.2), a total of 919 which measured (~1,38 ± 0,9 mm) in size. Most of them were transparent (34%) then black (27%), blue (19%) and green (8%). Yellow, pink and green fibres were also observed however due to their less quantity (10%) and frequency through the core, their distribution was not found to be significant. We also observed very few fragments around a total of 12 (2%) throughout the core, hence we exclude them in the analysis.

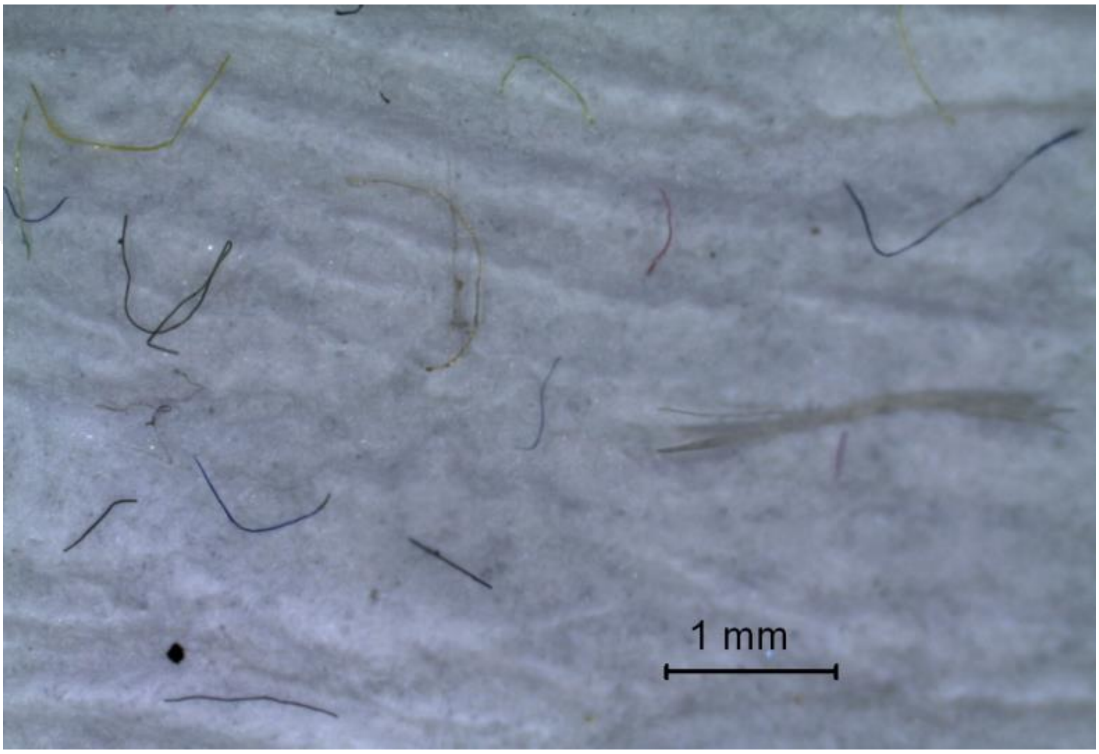

Figure 3.2: Example of fibre particles present in Uluabat sediment layer (Photo: Ali Serhan Çağan).

3.2. Core Chronology and Loss on Ignition (LOI)

The sediment around Kocacay Delta site was prone to frequent mixing and a high sedimentation rate (7,74 kg m^{-2} y^{-1} on average), consequently the core samples were dated only back to 2015 (Figure 3.3). While the core chronology of Kocacay Delta samples was rather recent, they however contained more varied microplastic types and colours and also higher microplastic content compared to that of Lake Uluabat. Following the LOI analysis at 550 °C, change was hardly observed similarly for carbonic content at 950 °C although there was a noteworthy carbonic content income increase from 2019 to 2020 (Figure 3.3).

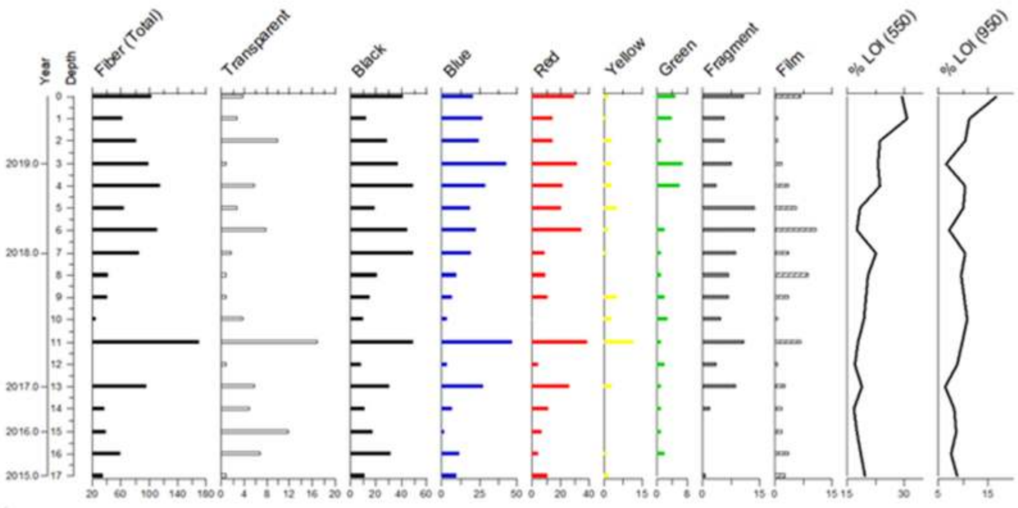

Figure 3.3: Distribution of microplastics through time and LOI values of sediment samples in Kocacay Delta.

When the core samples from Lake Uluabat were dated, it was found that microplastic accumulation in accordance with the innermost layers (30-35 cm) went back as far as 1966, while the outermost layer was dated to 2020 (Figure 3.4). The sedimentation rate of the Lake Uluabat was 2,52 kg $m^{-2}y^{-1}$ on average at the deepest point of the lake. Microplastic were distributed irregularly throughout the core. The microplastic distribution in the Lake Uluabat core were categorised samples into three zones; 1966 to 1996 (37-20 cm), 1996 to 2007 (9-11 cm) and 2008 to 2020 (8 cm- top most layer). An increase in trend of the microplastic accumulation was recorded for the first zone starting from as low as 9 particles per layer to 90 particles between the years of 1966 and 1996. In the second zone, the accumulation rate was recorded to be constant with most layers having approximately 28 particles. Interestingly, the amount of microplastics were observed to drastically increase in 2007 reaching up to 56 particles in that sediment layer. However, from that point onwards to the top most layers (2020) the accumulation rate decreased to an average of 29 particles per layer (Figure 3.4). There were no clear changes recorded for LOI analysis at 550 °C and carbonic content at 950 °C throughout the core, however a significant quantity of carbonic content income was observed through the years 2003 to 2008 (Figure 3.4).

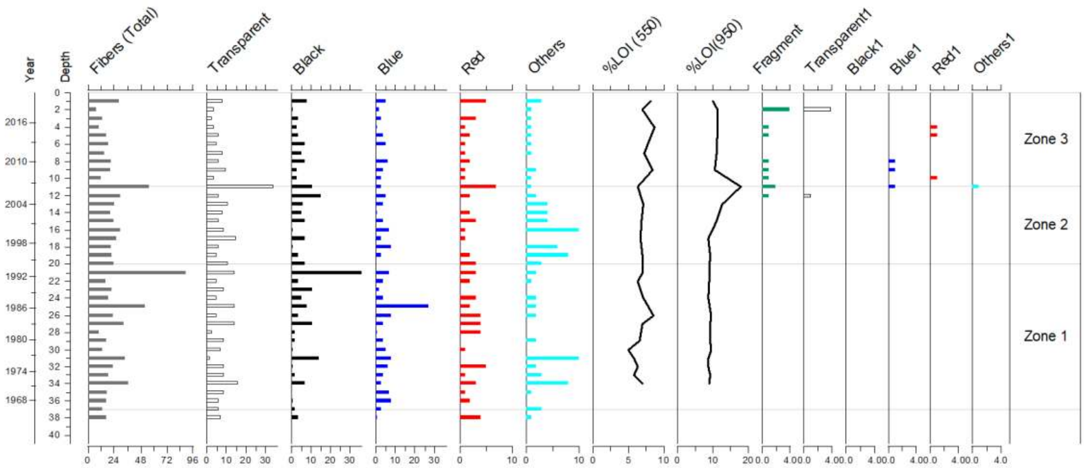

Figure 3.4: Distribution of microplastic through time and LOI values of sediment samples in Lake Uluabat.

4. DISCUSSION

Using a palaeolimnological point of view, we used microplastic as a proxy to trace plastic pollution in highly populated Bursa region. Kocacay Delta showed a higher microplastic abundance due to its exposed position while Lake Uluabat had less microplastic, the amount was still significant for a RAMSAR protected area. Despite, most water reserves in Turkey are exposed to pollution, drought and hydrologic impacts whilst having a rich biodiversity and high economic value (Coops et al., 2003; Beklioğlu et al., 2020; Coppens et al., 2020). Compared to research on marine environments, research on freshwater is moderately low (Sihicelli et al., 2018; Jian et al., 2020) even the exposure of pollution in both environments is evenly ubiquitous. Plastic pollution is currently a hot topic throughout the globe, unfortunately, studies revealing microplastic accumulation from past to present day are limited.

In the current study, the microplastic in the first zone of Lake Uluabat core (1966-1996) had an increasing tendency which coincided with the water level fluctuations of the lake recorded through 1984 to 1996 which could be attributed to low water level (Levi et al., 2016). Sediment accumulation rate may get higher during low water level seasons in shallow lakes (Kazancı et al., 2004; Reed et al., 2008) and a decrease in lake area was recorded from 1984 at 133,1 km^2 to 120 km^2 in 1993 (İleri et. al., 2014) which links up to the high microplastic abundance. The lake experiences extreme algal blooms from time to time due to the discharges from factories and agricultural water runoff (Magnin and Yarar, 1997; Karacaoğlu et al., 2004; Kazancı et al., 2004; Reed et al., 2008; Yurtseven and Randhir, 2020). The lakes nutrients are highly affected by organic and inorganic pollution affecting the eutrophication status of the lake. A monthly sample performed during 1998-1999 showed that the lake had high chlorophyll-a, nitrate and soluble reactive phosphorous concentrations (Dalkıran et al., 2006). These physico-chemical data however not directly connected with microplastic pollution but provide valuable information on the past pollution status of the lake. The euthrophic conditions could be as a result of the agricultural runoffs and industrial activities in the region and this would be associated on the inflow of microplastic into the lake. Later, a new status for the lake was designated when Lake Uluabat and its surrounding area was included in the

Ramsar List in 1998 (Çağırankaya, and Meriç, 2013). This new status made the lakes significance renowned at both national and international level as significant for the country and humanity together (Ramsar Convention Secretarial, 2010). These measures proved fruitful as they brought about a decrease in pollution status of the lake. Consequently, a decrease in microplastic numbers in the second zone through the years 1996 to 2007 was observed. A high income of inorganic/carbonate content at 950 °C was observed in 2007 coinciding with an increase in microplastic numbers. This may be attributed to higher urban and agricultural waste inputs to the lake resulting to water pollution (Bulut et. al., 2010) and the water level decrease in the lake recorded during this period (Degirminci et al., 2006). In addition, there is quite a few active drainage discharges around the lake shore besides the urban and industrial pollution that directly reaches the lake through Akçalar stream (Hacısalihoğlu and Karaer, 2020). In the third zone (2008-2020) microplastic numbers incessantly decreased. This may be because of the Uluabat Lake Sub-Basin Qualit Action Plan implemented by the Ministry of Agricultural and Forestry General Directorate of Water Management to help maintain its ecological importance and improve water quality (Ministry of Environment and Urbanization, 2020) which may have resulted to a decrease in amount of pollution reaching the lake during that period of time. Compared to the previous years, a remarkably high number of microplastic was detected at the surface sediment layer which corresponds to 2020. No literary data on waste influx into the lake is yet available for this period of time. However, during the Covid-19 pandemic lockdown microplastics may settle in the uppermost layer since there were no antropogenic sediment disturbances. Distribution of accumulating sediment is interrelated to lake bathymetry and basin morphology (Hilton et al., 1986), therefore, these dynamics may play a big role in controlling the deposition and re-suspension of microplastics in Lake Uluabat. Eutrophic lakes are also characterised with a high sediment accumulation rate which aids microplastic in mixing with organic matter increasing the sinking rate of microplastics (Porter et al., 2018). Additionally, effects like geological and geomorphologic structure of Lake Uluabat's basin, climate and vegetation, the lands steepness and water erosion also occur in the basin collectively with anthropogenic factors altogether bringing about an increase in the pollution load into the lake resulting to unfavourable effects to the water quality (Yenilmez and Aksoy, 2013).

In this study, the major microplastic particles from Lake Uluabat core samples were transparent, black and blue fibres. The findings are consistent with the other studies that fibres were the most observed microplastic particles in an urban lake sediment core (Turner et. al., 2019), a river sediment core (Horten et. el., 2017) and a marine sediment core (Woodall et. al., 2015). The main activity in the vicinity is fishing as in the highly populated Gölyazı village due to lack of agricultural lands, approximately 80-85% of the population has been fishing since the 1960s (Değirmenci et al., 2006). During the 1980s, fishing of crawfish was quite common, however due to a parasite infection; the population dynamics of crawfish was changed which resulted to a decrease of crawfish fishing after the 2000s (Yarar and Magnin, 1997; Çelik, 2000). As a consequence, the high abundance of fibre detected could be attributed to degradation of fishing nets before 2000s. Fishing has become less economical important due to pollution faced in the lake, majority of the population seek jobs in Bursa (Okumuş, 2019). Because of its historical nature, tourism has become the main economic practice in Gölyazı village however pollution persists from garbage dumps around the village which find their way into the Lakes (Okumuş, 2019). Approximately 60% of synthetic fibers are consumed globally and are mostly made of polyester, polyamide, acrylic polymers and polyolefins (Salvador Cesa et al., 2017). Despite we have no polymer composition data but the observed fibers would have originated from textile industry because textile is important economic sector in the region. Fibres are easily transported through atmospheric fall out and river run-offs hence have been reported in beaches, estuarine, marine, freshwater lakes, floodplains, intertidal mangroves, deep-sea areas and even in an artic lake (Van Cauwenberghe et al., 2015; González-Pleiter et al., 2020). Approximately, up to 1900 fibre particles were released per washing cycle from synthetic clothes which may end up in the environment through waste water treatment plants (Browne et. al., 2011; Salvador Cesa et. al., 2017). It has also been noted that the water quality of the main water inlets (Rivers Mustafa Kemal Paşa, Emet, and Orhaneli) are quite polluted. Therefore, they carry out high amounts of solid suspended material into the system in addition to organic and inorganic waste (Değirmenci et al., 2006) which could be a carrier of the lightweight and low density microplastics into the lake. For example, microplastic polluted discharges from Power Plants and several leather industry discharges enter into the Lake through

Orhaneli and Mustafa Kemal Paşa Streams (Akdeniz, 2011). The limit values of sediment quality regarding microplastic pollution should be urgently evaluated by both national and international standards (İleri et al., 2014).

Even though dating of the Kocacay Delta core was challenging, high amounts of microplastic were detected coming through the Susurluk basin into the Marmara Sea. The river system of the Susurluk basin has several branches thus, the variety of the microplastics (fibres, fragment, film/nylon) 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 in a considerable extent (Dalkıran et al., 2006). Coinciding with the current study, the sediments of a Marmara Sea shore close to the Kocacay Delta also detected a high number of fibres (Gürbüz, 2017).

5. CONCLUSION

Records of microplastic debris in sediments can help the researchers and policy makers to understand and evaluate microplastic pollution throughout history. The current study supported that microplastics are a useful proxy for paleolimnological studies to evaluate the historical anthropogenic impacts in freshwater environments. The accumulation of such emergent concern contaminants in both river and lake ecosystems can be detected successfully.

This information might help us to understand the impact of such pollutants to the ecosystem as well as showing the importance of controlling discharge waters to freshwater systems. Since the depletion of microplastic particles are still under debate and many scientific studies 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 into the environment again. In future studies, more core sampling would be performed from different points to evaluate the past plastic pollution where several places around the world have no data exist for the past accumulation. The polymer composition should take into consider through the core profile in the future studies.

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APPENDICES

APPENDIX 1. CURRICULUM VITAE

Name – Surname : Fatma Feisal AALMAS

Place and Date of Birth :

Address and Telephone :

Undergraduate : 2019, Kocaeli Universıty, Faculty of Arts and Sciences, Biology

Work Experiece:

Scientific Orgnisations:

Scientific Study Areas
Biology, Mikrobiology, Environment, Palaeolimnology

Publications:

Tatlısu ekosistemlerinde mikroplastiklerin çok yönlü değerlendirilmesi: Susurluk Havzası Ön sonuçları (2021). Ülkü Nihan TAVŞANOĞLU, Gökben Başaran KANKILIÇ, Belda ERKMEN, Gülçin AKCA, Ayhan FİLAZİ, Begüm Yurdakök DİKMEN, Gizem BEZİRCİ, Ali Serhan ÇAĞAN, Kerem GÖKDAĞ, Tamer ÇIRAK, Selin Özkan KOTİLOĞLU, Özgür KUZUKIRAN, Tülay PEKMEZ, Elif PAÇAL, **Fatma Feisal ALMAS** ve Melike SEYFE. 9.Ulusal Limnoloji Sempozyumu, 04-06 Ağustos 2021, Burdur. (Oral Presentation)

Scientific Activities:

Scholarships, Awards, Projects:
TÜBİTAK- ÇAYDAG project no: 119Y031

Other Informations: