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ORIGINAL PAPER

Microplastic contamination in freshwater: first observation in Lake Ulansuhai, Yellow River Basin, China

Zhichao Wang[1] · Yiming Qin[1] · Weiping Li[1] · Wenhuan Yang[1] · Qing Meng[1] · Jianlin Yang[1]

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Abstract
Microplastic pollution has been widely studied in the marine environment, but is much less explored in terrestrial waters, notably in China. Therefore, we studied the degree of microplastic pollution in surface waters of Lake Ulansuhai, a major freshwater lake in the Yellow River basin of northern China. Results show microplastic concentrations ranging from 1760 ± 710 to 10,120 ± 4090 n/m^3. The microplastic spatial distribution is heterogeneous, with higher levels near the drainage canal entrance of Lake Ulansuhai, and a downward trend from north to south in the lake. The main type of microplastics is colored particles, including fibers as the most abundant. More than 80% of microplastics were smaller than 2 mm. FTIR analysis results show that the main plastics were polyethylene, polystyrene and polybutylene terephthalate. There were also some metallic elements adsorbed on the surface of microplastics, such as Fe, Ca and Zn, detected by energy-dispersive spectrometry. The presence of metallic elements may worsen water pollution.

Keywords Microplastic · Contamination · Freshwater · Yellow River Hetao Irrigation District · Lake Ulansuhai

Introduction

Plastic products have brought great convenience based on their nature of durability, cheapness and light weight, the heavy using of them, but its difficulty of degradation have brought plastic waste a severe environmental trouble (Barnes et al. 2009; Wright et al. 2013). Plastic particle with size less than 5 mm was termed as microplastics, the source of it could be the smaller plastic particles (primary source) produced in the plastic manufacturing process or the plastic debris (secondary source) produced by the decomposition of large pieces of plastic (Frias et al. 2016). Since the word “microplastics” was first used in 2004, its distribution, types, and transfer path have attracted much attention (Thompson et al. 2004; Siegfried et al. 2017); even the *Nature* reported two successive studies on microplastics of marine floating and submarine sediment in 2014 (Marris 2014; Perkins 2014). Concerns to the existence of microplastics and its potential impact on ecological environment, even human health, have enhanced worldwide (Barboza and Gimenez 2015; Desforges et al. 2015).

Due to their unrestricted and ubiquitous characteristics, microplastic particles have been detected in marine, estuary, and even the Arctic (Desforges et al. 2014; Zhao et al. 2014; Lusher et al. 2015). Studies have shown that 80% of the microplastics in the ocean came from plastic products on land, and it was related to the surrounding enterprises, the distribution of residential areas and the unreasonable disposal of domestic waste (Derraik. 2002). The quantity of microplastics in ocean was at a severe degree and even reached 100,000 n/m^3 in some sea areas (Frias et al. 2014). Some studies for microplastic in ocean indicated that the particles could appeared negative effects for aquatic animals by ingesting and even produced a bad effect for human beings health through food web, based on its feature of small size. Researches on marine microplastics have found that the presence of microplastics in zooplankton, fish, even mammals (Cole et al. 2011; Bellas et al. 2016; Davidson and Dudas 2016). Rochman et al (2014) revealed the influence of the complex of microplastics and persistent organic pollutants on the endocrine system function of Japanese medaka (*Oryziaslatipes*).

Compared with the fruitful research on microplastics in marine worldwide, little information could be obtained for

✉ Weiping Li
sjlwp@163.com

[1] College of Energy and Environment, Inner Mongolia University of Science and Technology, Baotou 014010, China

the research about microplastics in freshwater lakes, especially in China. It has been reported that microplastic particles have been found in the Pearl River Estuary and even the lakeshore sediments in Tibet plateau (Fok and Cheung 2015; Zhang et al. 2016). The abundance, type and quantity of microplastics in water have been known by studies in the corresponding water bodies. Results of the study on microplastics in the Three Gorges Reservoir area showed that the amount of surface water microplastics in the region was relatively large, ranging from 1597 to 12,611 n/m^3. It also has been found that the amount of microplastics shows a significant correlation with water pollution factors, such as total phosphorus and chlorophyll a (Su et al. 2016). However, little information could be acquired on microplastics in rivers and high-salinity lakes of the Yellow River Basin in China.

Lake Ulansuhai was one of the largest freshwater lakes in northern China and was the largest wetland area in the Yellow River drainage basin (Kobbing and Niels 2016). Lake Ulansuhai was also a distinctive drainage area in Hetao Irrigation District, which was an important commodity grain production basis (Zhu et al. 2014). The prosperity of agricultural production in Hetao Irrigation District was closely related to the wide application of agricultural plastic mulching technology, and its usage quantity reached 21,000 tons in 2014 (Wang et al. 2014). Due to the wide application of agricultural plastic and the difficulty of its recycling, the small-sized plastics—one of the main sources of microplastics—degraded by weathering of agricultural film would enter the lake with the recession of farmland water (Zhang et al. 2017). Lake Ulansuhai was a rare large grassy lake with biodiversity and environmental protection function in desert and semi-desert areas worldwide, and it played a significant role in the global ecosystem and biodiversity. Hence, the research on microplastics of Lake Ulansuhai could provide a constructive suggestion and idea for the lake pollution control and water ecological restoration.

Materials and methods

Study area and sampling sites

The study area, Lake Ulansuhai, is situated in the largest wetland on earth at this latitude (40°47′–41°03′N, 108°43′–108°57′E), and the lake is located in Urad Qianqi, the Bayannaoer prefecture of Inner Mongolia Autonomous Region, China (Ruihong et al. 2004; Liu et al. 2017). As the eighth largest freshwater lake in China, Lake Ulansuhai has become the receiving water body of irrigation return flow, domestic sewage and industrial wastewater in Hetao basin (Ruihong et al. 2004; Shengnan et al. 2018; Zhu et al. 2014). Owing to its position, Lake Ulansuhai receives about 90% of the agricultural drainage in this area each year, and it is also a collection site for municipal wastewater and industrial wastewater from surrounding cities (Long and Jinglu 2010; Qi et al. 2018). Given that it exists as a sink for many contaminants, it is significant to investigate whether the contamination like microplastics existed in Lake Ulansuhai (Yan et al. 2017). Many factors should be considered for the selection of sampling points, such as specification of sampling, and hydrology water functional zoning of the lake. In this study, we have chosen nine representational sampling sites, and they were arranged at the entrance of the drainage channel to lake, the center and the end of the lake, respectively (Fig. 1a).

Sample collection and preparation

Sampling work was carried out in October 2018, and nine samples have been collected. The prototypes from each sampling point were taken in two copies for the accuracy of data, and position of sampling points was located by GPS (Global Positioning System). Briefly, twenty liters of surface water at depth 20 cm from the surface of the lake was collected by cleaned 12-V DC (direct current) Teflon pump and then filtered by a 48-μm stainless steel sieve, and material obtained on sieve was washed into clean glass bar. 5% formalin solution was added in it, and the samples were kept at 4 °C before laboratory analysis (Di and Wang 2018). For avoiding the possible negative effects of plastic, the use of plastic products during experiments was prohibited (Su et al. 2016). All water samples were soaked in 30% hydrogen peroxide solution and were placed in an oscillating incubator at 45 °C and 150 rmp/min for 24 h to digest organic and inorganic impurities. Samples were filtered by glass microfiber membrane with bore diameter of 0.45 μm (GF/F, 47 mmØ, Whatman). Samples were covered with tinfoil until detected for avoiding contamination of impurities from air. The filter was placed in clean petri dish at 50 °C, waiting for microscopic examination (Wang et al. 2017a, b; Klein et al. 2015).

Observation and identification of microplastics

Microplastics on filter paper were identified by stereo microscope (M165FC, Leica, Germany) and laser confocal microscopes (Olympus, OLS4000). Visual inspection was adopted to differentiated suspected microplastic particles and the method was mainly based on the physical characteristics of microplastics, referring to the classification standards created by previous studies (Hidalgo-Ruz et al. 2012). The unit for calculating the abundance of microplastics in water samples was n/m^3.

It was not sufficient and rigorous to distinguish and identify plastics only by stereo microscope and laser confocal microscopy (Eriksen et al. 2013). Hence, the FTIR

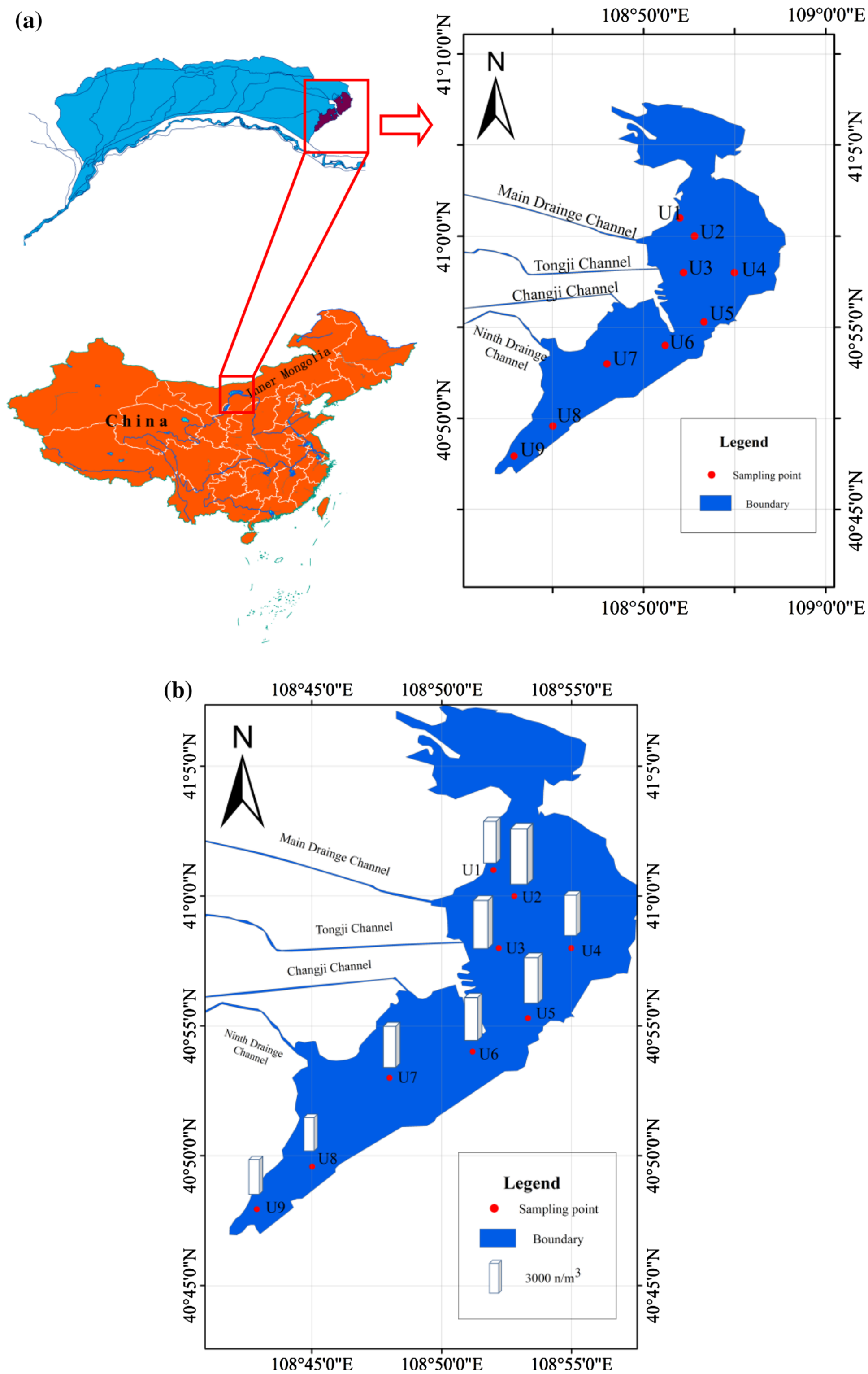


Fig. 1 Geographic position and water sampling points location of Lake Ulansuhai (**a**); abundance of microplastics in Lake Ulansuhai (**b**)

(Fourier transform infrared) spectroscope, SEM (scanning electron microscope) and EDS (energy-dispersive spectrometer) had been used for further analysis of microplastics. FTIR had been applied to analyzing the functional groups of microplastics for the ascertainment of their chemical structures (Thermo Fisher, Escalab 250Xi). Chemical composition of microplastics was awareness by comparing the obtained spectral data with database from the detection system. Due to the long immersion and hydraulic impact of Lake Ulansuhai, some mechanical scratches and periphery erosion occurred on the surface of plastic particles, so it was necessary to observe the surface of particles by SEM (Quanta 400, Fei, the USA). Before the samples were observed, the surfaces of each sample were covered with a gold film evenly, and the sample elements were determined by EDS.

Results and discussion

Abundances and spatial distribution of microplastics in Lake Ulansuhai

Numerous microplastics were detected in Lake Ulansuhai by this study and found in all sampling points ranging from 1760 ± 710 to 10,120 ± 4090 n/m^3. Compared with other studies about microplastics in freshwater lakes, the amount of it in Lake Ulansuhai resembled that in freshwater lake of Wuhan and Three Gorges Reservoir (Wang et al. 2016; Di and Wang 2018). Figure of microplastics in each point was different, which reflected the heterogeneity of its distribution.

Distribution of microplastics in Lake Ulansuhai presented a declining trend from U1 to U9, which revealed an exceptional spatial correlation between the microplastic particle in aqueous and the distance to the source of it (Fig. 1b). In particular, U2 had the crest value of microplastics at 10,120 ± 4090 n/m^3, followed by U3 at 9200 ± 4220 n/m^3 and U1 at 8080 ± 3150 n/m^3. These three sampling points were all located in the northwest of Lake Ulansuhai, which also were receiving positions of main drainage channel and Tongji channel. These two backwater drains were the main path of farmland recession, wastewater from industrial and domestic sewage from cities and towns surrounding the lake (Liu et al. 2017). Intensive human activities were the most important reason for the rapid increase in microplastic pollution in lakes (Eriksen et al. 2013). Agricultural plastic film used in planting could not be recovered effectively; studies have shown about 30% of agricultural film residues in soil in Hetao Irrigation District every year, and large proportion of the remaining part was carried into Lake Ulansuhai in the process of returning water to it (Wang et al. 2017a, b). This was an indispensable source for the accumulation of microplastic pollution in Lake Ulansuhai. Domestic sewage entering the lake through drainage channels was another important source of microplastics, and the amount of microplastics could be increased by cosmetic abrasive particles and plastic fibers originated in the washing process (Fendall and Sewell 2009). Amount of microplastics in sampling sites U5, U6 and U4 was not severe as above, which was 4120 ± 1270 n/m^3, 3720 ± 1370 n/m^3, and 3020 ± 1160 n/m^3, respectively. Although there was drainage water inflow in the lake through Chang ji Chanel near U6 site, the bulk lake water produced a dilution effect, and the same effect was more evident at U4. There was no a large-scale retreat canal near the sites U7 (3200 ± 1250 n/m^3), U8 (1400 ± 390 n/m^3) and U9 (1760 ± 710 n/m^3); hence, the concentration of microplastics at these points decreased further by dilution and the diffusion of the entire region. Notwithstanding, the values of these points were smaller than others in Lake Ulansuhai, whose number was also relatively large. It must be mentioned that a large amount of plastic waste thrown into the lake, such as plastic bags and beverage bottles, may be an important contributor for microplastics in the lake, and residual fishing nets in the fishing process also had a positive impact on the increase in plastic particles in it (Zhang et al. 2016; Qian and HaiBo 2016). As rivers and lakes would eventually merge into the ocean, the microplastics contained in the water bodies may also enter the ocean. Data in Appendix Table 1 show that the content of microplastics in the ocean was much lower than that in lakes, indicating that microplastics may eventually be transported to the ocean along the flow of rivers.

Morphological characteristics of microplastics in Lake Ulansuhai

Main shapes of microplastics

Fiber, film, fragment and grain were detected in Lake Ulansuhai; the typical image of them by laser scanning confocal microscope and the percentage histogram are shown in Figs. 2a and 3a, respectively. Types of figure of microplastics were commonly reported in urban surface water of Wu han (Wang et al. 2016) and surface water of the Laurentian Great Lake (Eriksen et al. 2013). In all shapes of the forms, fiber was the largest category, occupied from 68.18 to 78.64%. Studies have shown that domestic sewage containing fabric fibers produced by washing machines during washing was an important source of microplastics in lakes (Browne et al. 2013). The proportion of fragment in surface water was 5.4%; evenly, fragment could originate in many aspects, such as the crushing of plastic packaging materials, plastic containers and clean media (Thompson, et al. 2004). In Lake Ulansuhai, the agricultural film brought in it by the farmland retreatment may produce a certain amount of fibrous microplastic under the action of long-term hydraulic erosion, which may contribute to the increase in the number of microplastics, and based on this, the ratio of thin film microplastics was relatively large. Proportion of grain in the lake was 2.6–11.4%. Granular plastics were important raw materials for the production of plastic products, such

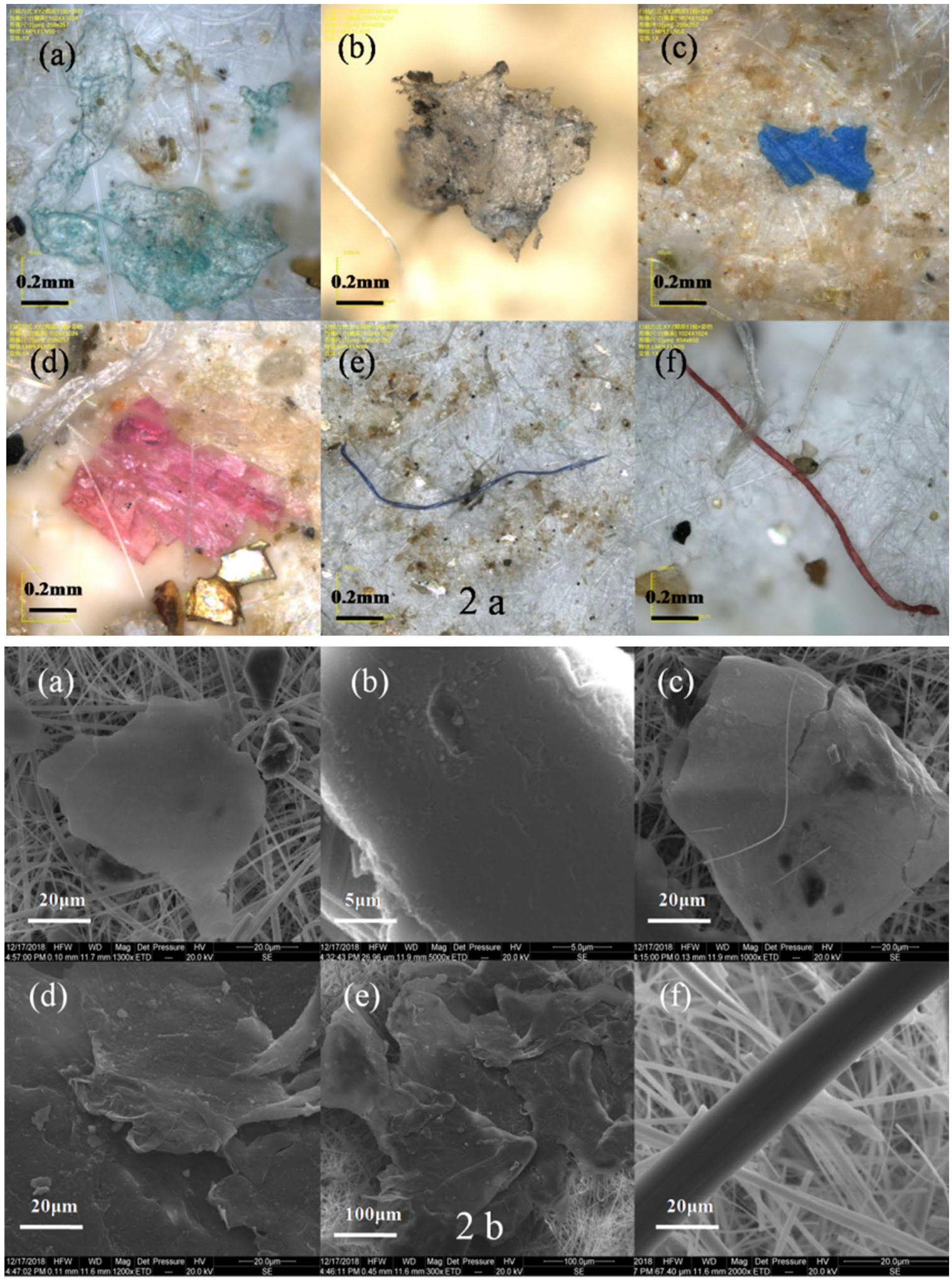


Fig. 2 Microscopic (**a**) and scanning electron microscopy (SEM, **b**) images of microplastics in Lake Ulansuhai. There are films (2a.a, 2a.b), fragments (2a.c, 2a.d), and fibers (2a.e, 2a.f) with different colors and sizes. SEM image of fragments (2b.a, 2b.b, 2b.c), films (2b.d, 2b.e) and fibers (2b.f). These graphs describe the surface appearance and the degree of damage due to hydraulic friction of microplastics

as containers and plastic sheets, and granular plastics produced in domestic waste also contributed to the growth of its quantity (McDermid and McMullen 2004). However, the exact source of these plastics was not clear, and further studies and analysis were needed.

Main color of microplastics

Colored plastic particles account for 71.5–94.9% in the lake (Fig. 3b). It could be seen that color microplastics were the main contamination in Lake Ulansuhai. And there were many types of colors, including black, red, blue, green, magenta and transparent. In the process of obtaining water samples, the existence of discarded water bottles was found in several parts of the lake. Colored plastic granules were themselves a water pollutant, and the dyed organic matter attached to it was also at risk of contamination, which may have potential toxic effects on aquatic animals (Hämer et al. 2014; Browne et al. 2013). Transparent plastic had a percentage of 5.1–21.5% in surface water. Plastic film mulching technology had obvious functions of increasing temperature and preserving soil moisture, and

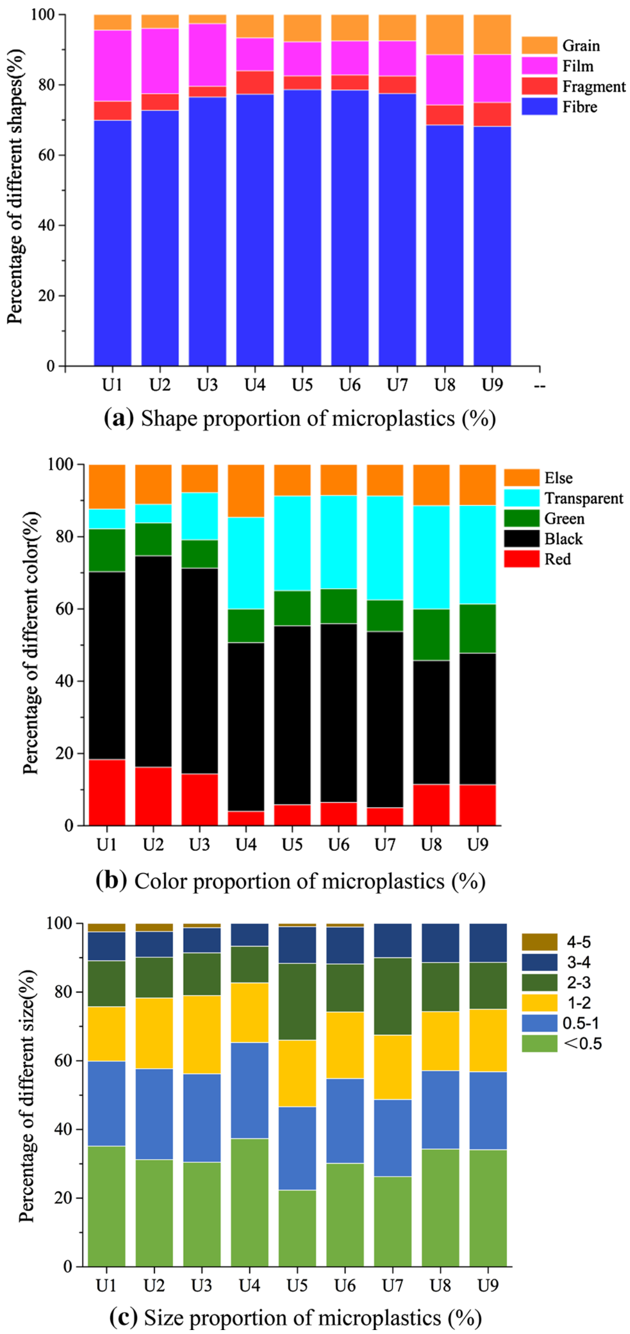


Fig. 3 Shape (**a**) color (**b**) and size (**c**) proportion of microplastics in Lake Ulansuhai. It depicts the variousness of shape, color and size in all sampling sites and percentage disparity in each site

it has been widely used in arid and cold areas of North China (Wang et al. 2017a, b). Agricultural film mulching technology had extensive application in Hetao Irrigation District China, and its usage quantity had reached 21,000 tons by 2014 (Wang et al. 2017a, b). Based on this, unrecovered agricultural residual film may be one of the important sources of transparent microplastics.

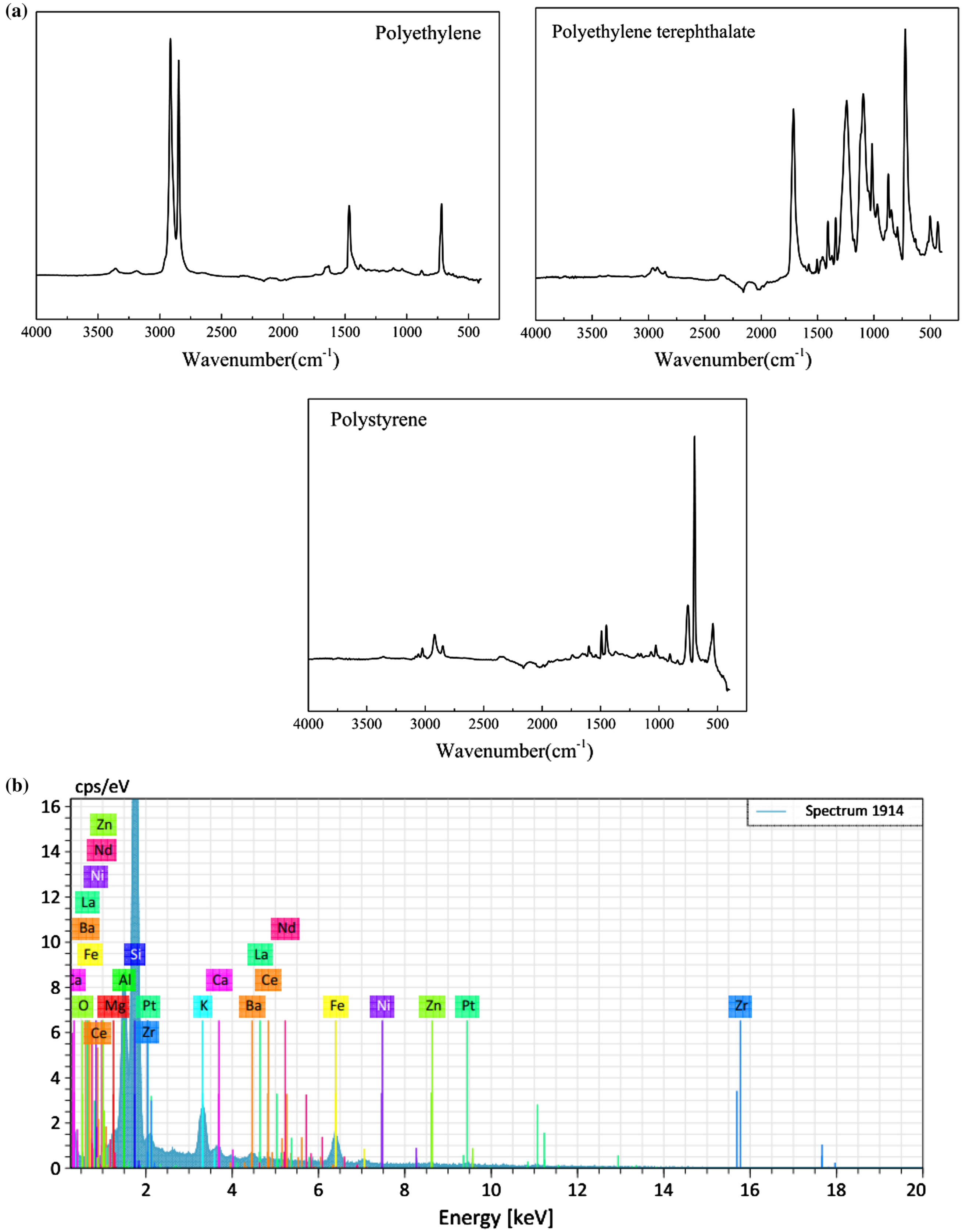
Fig. 4 Fourier transform infrared spectroscopy (FTIR) images of microplastic samples in Lake Ulansuhai (**a**) and energy-dispersive spectrometry (EDS) images of microplastics samples in Lake Ulansuhai (**b**). It depicts the chemical composition of main types of microplastics by representative chemical bond and the existence of metal elements on its surface ▶

Main size of microplastics

In this study, the microplastics of Lake Ulansuhai were divided into six categories according to their sizes: < 0.5 mm, 0.5 mm–1 mm, 1–2 mm, 2–3 mm, 3–4 mm and 4–5 mm. As could be seen from Fig. 3c, the quantity of plastics smaller than 2 mm accounted for a large proportion, and the average proportion was 74.74%. Analogous outcomes were reported in northern Gulf of Mexico estuaries (Wessel et al. 2016) and the Laurentian Great Lakes, USA (Eriksen et al. 2013). The results showed that there was a negative correlation between the quantity of microplastics and its size; that is, the smaller the size, the higher the content (Doyle et al. 2011). Combined with the previous analysis and related research, it could be concluded that under long-term immersion and hydraulic corrosion and mechanical friction, the plastic would be broken, peeled into small pieces and then converted into smaller plastics, such as fibrous microplastics lower than 2 mm, the advantage category in Lake Ulansuhai (Foekema et al. 2013). Small-sized plastic particles were harmful to aquatic organisms, because their size was close to zooplankton or other aquatic animals. When microplastics were eaten by mistake, they may have negative effects on aquatic organisms and then may harm human beings through food web (Cole et al. 2015; Su et al. 2016). There were few studies on the intake of microplastics by aquatic organisms, and further studies were needed.

Polymer validation of microplastics in Lake Ulansuhai

It was important to analyze the composition of substances, and identification of components of the microplastics was accomplished by FTIR. Polyethylene (PE), polystyrene (PS) and the polybutylene terephthalate (PET) were the main categories of plastic particles in Lake Ulansuhai, and their proportions were 63.7, 21.5 and 14.8%, respectively (Fig. 4a). PE was a common type of plastic in various sampling points, and as a major component of microplastics, it also appeared in other studies (Imhof et al. 2013). PE plastics were widely used in daily life for their characteristics of high lightness and light weight, such as packaging bags and containers, and they became the main source of microplastics by degrees (Sruthy and Ramasamy 2017).


(a)
Polyethylene
Wavenumber(cm-1)
Polyethylene terephthalate
Wavenumber(cm-1)
Polystyrene
Wavenumber(cm-1)
(b)
cps/eV
Spectrum 1914
Zn
Nd
Ni
La
Ba
Fe
Si
Ca
Al
O
Mg
Pt
Ce
Zr
K
Energy [keV]

Based on this, the agricultural film entrapped into the Lake Ulansuhai during the process of farmland dewatering was a source of a large number of PE microplastics. PS was widely used in the packaging of large items and thermal insulation board of buildings (Vianello et al. 2013), and PET was commonly employed as beverage bottles. The main sources of PS and PET may be the effluent of surrounding domestic sewage and the plastic that floats in the air and then falls into the lake. Compared with related studies, such as Taihu Lake (Su et al. 2016) and the Yangtze River (Zhao et al. 2014), the types of Lake Ulansuhai polymers were relatively small, so the research on the types of microplastics in the lake was needed for a further implementation.

Pollution adsorbed by microplastics in Lake Ulansuhai

Heavy metal contamination was detected on the surface of microplastics by means of EDS (Fig. 4b). It could be concluded that there were many metal elements adhering on the surface of microplastics, such as iron, zinc and nickel. Some rare earth elements, such as cerium and lanthanum, have also been detected. Pollution of metals in Lake Ulansuhai has been explained, there were abundant mineral resources in Lake Ulansuhai basin, and about 2×10^8 m^3 industrial wastewater was discharged into the lake per year, which resulted in the accumulation and pollution of heavy metals in the lake (Shengnan et al. 2018). The combination of heavy metals and microplastics may aggravate the harmful effect that exists for a long time, based on the unique characteristics of hard to degrade and suspension in lake. Studies on the adsorption of heavy metals by microplastics showed that it had a strong adsorption effect to metal elements; with the passage of time, heavy metals accumulated on their surface could had an exchange action with water (Brennecke et al. 2016). After a long time of hydraulic erosion and friction, the surface of plastic could produce notch and groove, which brought smaller plastic particles, also increased the specific surface area of it and offered more places for metal residence; the SEM image of microplastics in Lake Ulansuhai is shown in Fig. 2b. Nevertheless, the quantitative and mechanism studies on the combined pollution of microplastics and heavy metals and their biological toxicity have not been carried out, which required more attention and further research.

Conclusion

We assessed the pollution of microplastics in Lake Ulansuhai. Quantitative distribution showed an obvious spatial difference from the entrance of farmland drainage canal to the end of drainage canal of the lake. Colored plastic fibers with sizes less than 2 mm were dominated, and all these were inseparable with the residual agricultural film brought in by the farmland drainage in the region and the high-intensity human activities around it. The presence of metal elements was also detected on the surface of it, indicating that microplastics may have other contamination risks, but it was sealed. We need to pay more attention to the pollution of it and other biological toxic hazards it may bring to aquatic animals and plants, even human beings. Furthermore, our work provides a meaningful reference for the future research of microplastics in Lake Ulansuhai.

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Appendix

See Table 1

Table 1 Size and abundance of microplastics in some ocean and lakes

Investigation area	Main particle size	Abundance	References
Northeastern Pacific Ocean	558 ± 521 µm	1710 ± 1110 n/m^3	Desforges et al. (2014)
Austrian Danube	< 20 mm	0.32 ± 4.67 n/m^3	Lechner et al. (2014)
North Western Mediterranean Sea	0.3–5 mm	0.12 n/m^2	Collignon et al. (2012)
Southern coast of Korea	100–200 µm	23 ± 20 n/L	Song et al. (2014)
Yangtze Estuary of China	0.5 mm-5 mm	0.167 ± 0.138 n/m^3	Zhao et al. (2014)
Laurentian Great Lakes	0.355–0.999 mm	43,000 item/km^2	Eriksen et al. (2013)
Three Gorges Reservoir, China	< 1 mm	1597–12611 n/m^3	Di and Wang (2018)
Lake Hovsgol, Mongolia	0.333–4.749 mm	20,264 n/km^2	Free et al. (2014)
Lakes in Tibet plateau, China	< 5 mm	563 ± 1219 items/m2	Zhang et al. (2016)
Taihu Lake, China	100–1000 mm	3.4–25.8 items/L	Su et al. (2016)

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