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Microplastic Accumulation in Agricultural Soils with Different Mulching Histories in Xinjiang, China

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Abstract: Microplastics (MPs) in soils have been widely studied, yet very little is known about their distribution in agricultural soils and the impact of mulching history. In this study, soil samples were taken across 3 soil layers of 60 sites with varying years of mulching history (<5 years, 5–10 years, 10–20 years and >20 years) in agricultural film-mulched cotton fields of Xinjiang, China. Microplastics were obtained from the soils using oil separation combined with density separation. Stereomicroscopy and Fourier transform infrared spectroscopy (FTIR) were used for identification. The average microplastic abundance of the sites with different years of mulching history are 538, 1484, 5812 and 9708 pieces/kg, respectively. The microplastics with sizes 1000–5000 and 200–500 µm are dominant in soils with less than 10 years and over 10 years of continuous mulching history, respectively. The results show that the abundance of microplastics increases and the size of microplastics decreases gradually as the number of years of mulching history increases. In addition, the best polynomial fitting curves were found between microplastic abundance (y) and mulching years (x) in different soil layers, and the relationship in the topsoil layer can be fitted as the following equation: $y = 20.6x^2 = 41.39x + 198.65$ ($p < 0.01$, $R^2 = 0.62$). The results indicate that residual agricultural mulching film is the dominating source of microplastics in cotton fields. This study provides rationale for further research on microplastics prediction in agricultural film-mulched fields.

Keywords: cotton field; mulching; microplastic pollution; agriculture; soil; distribution; accumulation

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1. Introduction

Since the last century, plastic pollution has been one of the essential environmental problems in global ecosystems [1]. With the rapid increase in social and economic development, the world plastic production has increased and reached 367 million tons in 2020, almost 32% of which originated in China [2–4]. Small pieces of plastic, whether intentionally included or through the degradation of plastic products, are ending up in the environment [5]. Microplastics (MPs), which are defined as plastic particles less than 5 mm in length [6], are of great concern globally as environmental pollutants [7]. Some studies have shown that microplastics are widely distributed around the world, in the ocean [8], on land [9], on islands with lower human activity [10], and even in polar glacier [11]. Thus, microplastic pollution has been recognized as a serious global environmental issue.

Since 2010, the number of studies on microplastics has increased rapidly and continuously [12]. The relative research on microplastics started in the ocean, and then gradually turned to rivers and lakes on land. The study area included the northwestern Mediterranean Sea [13], the east coast of the U.S.A. [14], the mid-west Pacific Ocean [15], and some rivers [16,17]. Recent studies show that microplastics also represent an important threat to terrestrial ecosystems [18,19], but there are still few studies on the abundance and distribution of microplastics in soils. Soil is the initial gathering place for microplastics and annual plastic release in soil is 4–23 times greater than in oceans [20–22]. Zhang [23] found that average plastic particles in southwestern China's soil was 18,760 particles kg^{-1}, and most, 95%, are in the microplastic size range. Corradini [24] found that the level of microplastics in agricultural soil was 0.6–10.4 p g^{-1} and sludge is the main driver of soil microplastic pollution in Chile. Scheurer [25] found that microplastics in Swiss floodplain soils was 593 pieces·kg^{-1}; however, recent research lacks an exploration of the microplastic characteristics in soils with different years of mulching history, and the relationship between microplastics in different soil layers and mulching history were not well understood.

Agricultural production is the key to national economic development and security [26]. However, the microplastics in agricultural soil can be readily ingested by soil organisms, threatening crop growth and reproduction [27]. Furthermore, microplastics can even accumulate in the food chain and cause damage to soil biota at different trophic levels and entire ecosystems [19,28]. Thus, more attention should be paid to agricultural soil microplastic pollution in terrestrial ecosystems. The main polymer types of microplastics in soils includes polypropylene (PP), polyethylene (PE), polystyrene (PS), polyester fibers (PES), ethylene-propylene copolymer (EPC), polyurethane (PU) and others [29].

Xinjiang Uygur Autonomous Region is the largest cotton production base in China. In 2020, The planting area of cotton in Xinjiang was 2.5×10^6 ha [30], and was identified as a potential "hotspot" of microplastic contamination due to its long history of agricultural mulching films [31]. Mulching film, which was made from PE, has been widely used for crops in modern agriculture. Owing to a low recovery rate of the mulching film, the pollution of residual mulching film in agricultural soil is a serious problem in Xinjiang. A previous study showed that the cotton fields accounted for 75.7% of the total residual mulching film in Xinjiang [32]. Due to long-term photolytic, mechanical, and biological degradation, much of the residual mulching film turns into a microplastic pollutant [33–35]. Long-term mulching of agricultural fields leads to the accumulation of microplastics in the soil. Furthermore, the vertical distribution of microplastics in soils is inconsistent because microplastics can migrate through leaching, bioturbation and mechanical disturbance [36]. However, relevant research focused more on surface soil layers (<30 cm) [29,37], and deeper soil layers were neglected. Moreover, the distribution characteristics of microplastics in soils may be affected by many factors, such as mulching film history, irrigation methods, soil texture and so on [29]. Previous studies mainly investigated the microplastic abundance and characteristics, and microplastic-related topics of terrestrial ecosystems in arid regions with agricultural soils are still inadequate; therefore, whether the mulching history or soil depth affect microplastic distribution in such regions is yet to be investigated.

Corresponding research focusing on the topics mentioned above is urgently required. This study investigated the microplastic occurrence of residual mulching film in cotton fields in Xinjiang Uygur Autonomous Region, northwest China. This area is an important cotton base and mulch is used most years. The study aimed to reveal the relationship between different years of mulching history, different soil depths and the microplastic distribution in the soil of Xinjiang cotton fields.

The specific objectives were: (a) to reveal the effect of mulching history and soil depth on the distribution of soil microplastics; (b) to determine the effect of mulching history and soil depth on the characteristics of soil microplastics; and (c) to explore the relationship between microplastic abundance and mulching history. The study results are expected to provide valuable information for estimating and monitoring microplastic pollution in agricultural soils.

2. Materials and Methods

2.1. Study Area and Sample Collection

This study selected Tiemenguan City (Figure 1), where large areas of concentrated plastic mulching farmlands for cotton growing are located. Tiemenguan City has a total area of 590 km^2 and is located south of Tianshan Mountain and east of Tarim Basin. Cotton is a staple cash crop in this area, accounting for >50% of the total sown crop area. Polyethylene films have been widely used in this area over the past 30 years. The amount of polyethylene plastic mulching is 68 kg/ha, and the thickness of the film is 8 µm.

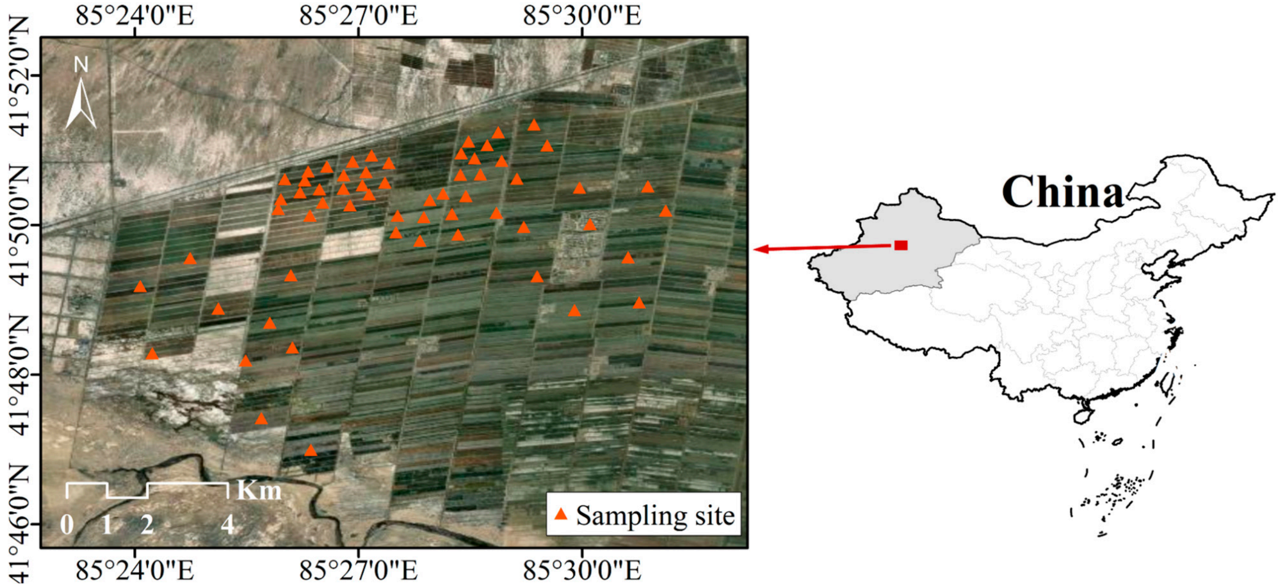

Figure 1. Location of the study area and sampling sites.

To investigate the effects of mulching history on the distribution of microplastics, samples from 60 sites covering different years of mulching history, including 0–5 years (n = 9), 5–10 years (n = 21), 10–20 years (n = 10) and over 20 years (n = 20), were collected from cotton fields in April and May 2021. The detailed data of mulching history was obtained through a local investigation and visits. At each sample site, three soil samples were collected at different depths, including the surface (0–25 cm), middle (25–35 cm) and deep (35–60 cm) soil layers. Therefore, there are 180 composite soil samples in total. The soils were collected with a soil auger, and a stainless-steel ruler was used to confirm the soil depth. Four subsamples were collected randomly and mixed evenly to constitute a composite sample corresponding to each soil layer, then they were stored in glass jars or paper bags. Eventually, the soil samples were transported to the laboratory for further analysis.

2.2. Sample Pretreatment and Microplastic Quantification

Soil samples were simply rolled and loosened, then air dried for 3–5 days. Large pieces of impurities and litter in soil samples were manually picked out. There was no need to sieve samples to avoid losing microplastics that wrapped around large aggregates. Microplastics were extracted from soils by combining density separation [38,39] and oil separation [40]. In brief, 0 g of air-dried soil was put into a custom-made 100 mL polytetrafluoroethylene centrifuge tube, and 25 mL of saturated sodium chloride solution and 3 mL of olive oil were added into the tube. The tube was placed in an ultrasonic cleaner (25 °C, 40 kHz) for 20 min and spun using a centrifuge at a speed of 5500 min^{-1} for 10 min. Adding the solutions, ultrasonic cleaning and centrifugation were repeated three times, but the last two ultrasonic cleanings only took 10 min, and olive oil was replaced with n-hexane on the third time. The supernatants after the three centrifugations were collected into a glass container, and 30 mL of n-hexane was added and stirred. After the mixed solution was filtered using a vacuum filtration unit, organic matters in the mixture which were obtained on the filter membrane (Millipore, 20 µm pore size, 47 mm diameter) were digested by Fenton's reagent [41]. Next, the microplastics were collected by filtering and transferred to glass Petri dishes. At last, they were dried in an oven at 38 °C and stored for subsequent optical inspection.

All the microplastics on the membrane filters were identified using the stereo microscope (50×–100×). They were photographed by a digital color camera equipped with the microscope. Uncertain microplastic-like particles were examined using a Fourier-transform infrared spectrometer (Thermo, Waltham, MA, USA, Nicolet 6700) in the mid-IR transmission mode at a resolution of 4 cm^{-1} with 16 scans for each spectrum in a spectral range from 4000 to 400 cm^{-1}. The FTIR spectra were compared with a reference database to determine the polymer type. The size of the microplastics was measured on the longest dimension of the image via Image J software. According to the size of the microplastics, they were divided into four categories: <200 µm, 200–500 µm, 500–1000 µm and 1000–5000 µm. They were then classified according to their size and quantity. The microplastic abundance of the soils was represented by the particle number (pieces/kg).

2.3. Quality Control and Quality Assurance

Contamination was strictly avoided for the duration of the experiment, and materials that could release microplastics were discarded, including clothing and sampling tools [42]. The sampling tools and consumable materials were rinsed with ultrapure water (18.2 MΩ) before use. In order to prevent microplastic contamination from the atmosphere, the containers were always covered with aluminum foils. Blank samples (ultrapure water without microplastics) were set up and run in parallel with real soil samples in the laboratory throughout the process to identify any ambient contamination [23,43]. The measured results showed that there was no contamination in the blank samples. In order to ensure the accuracy and reliability of the data, each sample was replicated three times [29,44]. Furthermore, the heat deflection temperature of polyethylene polymers ranges from 40 °C to 82 °C, so the working temperature was controlled to be below 40 °C to protect the microplastics from deformation.

For the determination of the microplastic recovery rates, 42 clean soil samples of 10 g were selected after the removal of all visible litter and plant residues [45], and test MPs (dimensions in the range 100–5000 µm) were spiked with the soil samples [46]. Each mixture was pretreated by the same method as real soil samples, and the related recovery experiment for each sample was conducted in triplicate. The results showed that the mean recovery rate of microplastics was 96.5%. Obtained after batch experiments using real soil samples, this method required only 4.5 h to separate microplastics from 54 soil samples.

2.4. Statistical Analysis

Differences in the abundance and size of microplastics under different soil layers and years of mulching history were determined using a one-way analysis of variance with the post-hoc Bonferroni test when the normality and homogeneity of variance of the data sets were satisfied. The results were considered significant at a p-value < 0.05. The microplastic abundances for different soil layers with a mulching history were analyzed by the polynomial fitting method in the Origin (Version 2021, Origin Lab) software. The best fitting equation was chosen by the equation with the highest determination coefficient.

3. Results

3.1. Distribution of Microplastic Abundance

3.1.1. Microplastics in Different Soil Layers

The abundance of microplastics was detected in cotton fields in the Xinjiang Uygur Autonomous Region. Microplastics can be found throughout the 60 cm plough layer. The average abundances of microplastics in the 0–25, 25–35 and 35–60 cm layers with a mulching history of under 5 years were 586, 557 and 471 pieces/kg, respectively (Figure 2A); and for a mulching history of 5–10 years, they were 1767, 1433 and 1252 pieces/kg, respectively (Figure 2B). There was no significant difference ($p > 0.05$) among the abundance of microplastics in the different soil layers under the two mulching film histories mentioned above.

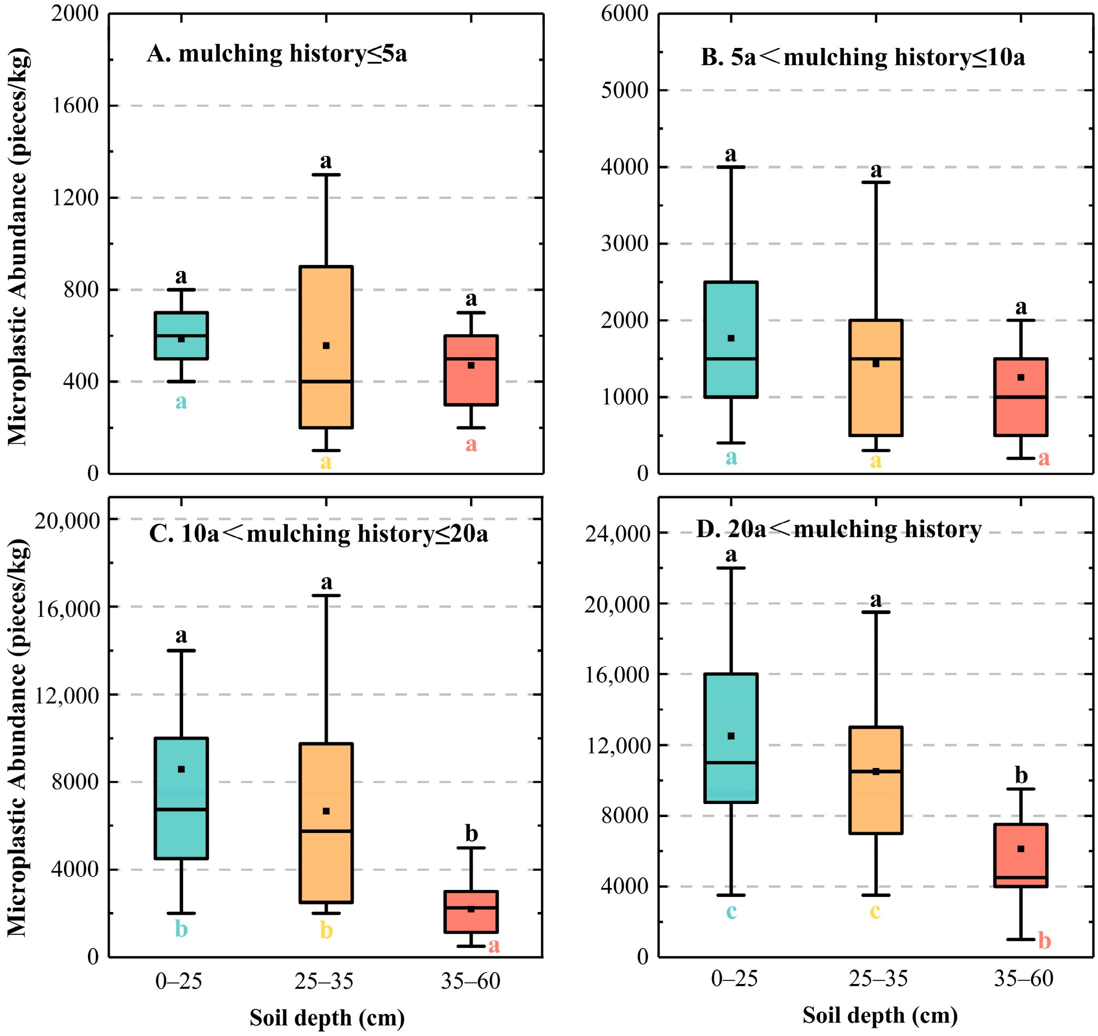

Figure 2. (**A**–**D**) The abundance of microplastics in different soil depths and with different mulching histories. Difference color columns represent different soil depth. Box boundaries indicate the 25th and 75th percentiles; black line and dot within the box: median and mean, respectively; whiskers below and above the box: minimum and maximum values, respectively. Difference lowercase with black and color letter show differences among different soil depths and mulching history respectively ($p < 0.05$).

In the field with 10–20 years of mulching, the average abundance of microplastics were 8583, 6667 and 2187 pieces/kg in the 0–25, 25–35 and 35–60 cm layers, respectively (Figure 2C). In the fields with over 20 years of continuous mulching, the average abundances of microplastics were 12,500, 10,500 and 6125 pieces/kg, respectively (Figure 2D). Under the two mulching histories above, the abundance of microplastics in the 35–60 cm soil layer was significantly lower ($p < 0.05$) than that of the 0–25 and 25–35 cm soil layers, and there was no significant difference in the abundance of microplastic between the 0–25 and 25–35 cm soil layers. Overall, it appears that the amount of microplastics in cotton fields decreased significantly with the deeper soil layers. Based on surveys of local farmers, the depth of tillage was mainly focused on shallower soil layer, which may account for the lower abundance of microplastics in deeper cotton fields soil.

3.1.2. Abundance of Microplastics with Different Mulching History

The average abundances of microplastics in film-mulched cultivated land with a history of less than 5 years, 5–10 years, 10–20 years and more than 20 years were 538, 1484, 5812 and 9708 items/kg, respectively. The results appear to show that the average abundance of microplastic in the whole soil layer increased significantly with mulching history. The similar increasing trends were in both the 0–25 and 25–35 cm soil layers.

As shown in Figure 2, the abundances of microplastics in fields with under 5 or 5–10 years of continuous mulching were significantly lower ($p < 0.05$) than the abundances of microplastics in fields with 10–20 years and over 20 years of continuous mulching. Furthermore, there was no significant difference ($p > 0.05$) in the abundance of microplastic in fields with below 5 years and 5–10 years of continuous mulching. It appears that the abundance of microplastics in fields with over 20 years of continuous mulching was significantly higher ($p < 0.05$) than other mulching years. In addition, as for the abundance of microplastics in the 35–60 cm soil layer, there was no significant difference ($p > 0.05$) in fields with under 5, 5–10 and 10–20 years of continuous mulching. Only the film-mulched years longer than 20 years had a significantly higher ($p < 0.05$) microplastic abundance than other mulching histories.

3.2. Shape Characteristics of Microplastics in Agricultural Soil

The size distribution of microplastics differed among the three soil layers, and it also varied with mulching history (Figure 3). The maximum average size of microplastics appeared in the field where the 35–60 cm soil layer was continuously film-mulched for less than 5 years, with an average size of 1523 µm. In contrast, the minimum average size occurred in the 0–25 cm soil layer with a mulching history of 10–20 years, with an average size of 578 µm. When the mulching history was less than 5 years, there was no significant difference ($p > 0.05$) in the size of microplastics among the three soil layers. It was consistent with the mulching histories of 5–10 years and over 20 years. Furthermore, when the mulching history was 10–20 years, microplastic sizes in 25–35 and 35–60 cm soil layers were both significantly higher ($p < 0.05$) than in the 0–25 cm soil layer; and, there was no significant difference in the size of microplastics in the 25–35 and 35–60 cm soil layers.

The average sizes of microplastics in film-mulched cultivated land with a history of less than 5 years, 5–10 years, 10–20 years and more than 20 years were 1374, 1402, 701 and 641 µm, respectively. The average sizes of microplastics in the entire soil layer showed a decreased trend with years of mulching history. Results showed that the sizes of microplastics in fields with a mulching history of 10–20 years and over 20 years were both significantly lower ($p < 0.05$) than the sizes of microplastics with 0–5 years and 5–10 years of continuous mulching (Figure 3).

The percentages of microplastics in the four size ranges in the whole soil layer at different cover histories are as follows: (1) mulching history $\leq$ 5 years: 1000–5000 µm (39.2%), >200–500 µm (24.6%), >500–1000 µm (23%), >0–200 µm (13.2%); (2) 5 years < mulching history $\leq$ 10 years: 1000–5000 µm (40.3%), >200–500 µm (27.2%), >500–1000 µm (20.7%), >0–200 µm (11.8%); (3) 10 years < mulching history $\leq$ 20 years: 200–500 µm (35.8%), >500–1000 µm (21.8%), >0–200 µm (21.4%), >1000–5000 µm (21%); (4) mulching history > 20 years: 200–500 µm (42.7%), >0–200 µm (23.9%), >500–1000 µm (17.2%), >1000–5000 µm (16.2%). The results showed that microplastics in 1000–5000 µm and 200–500 µm size were predominant in the fields with less than 10 years of continuous mulching and over 10 years of continuous mulching, respectively. The comparison between the different sections of Figure 4 indicates that the longer the mulching history, the greater the proportion of microplastics of small sizes.

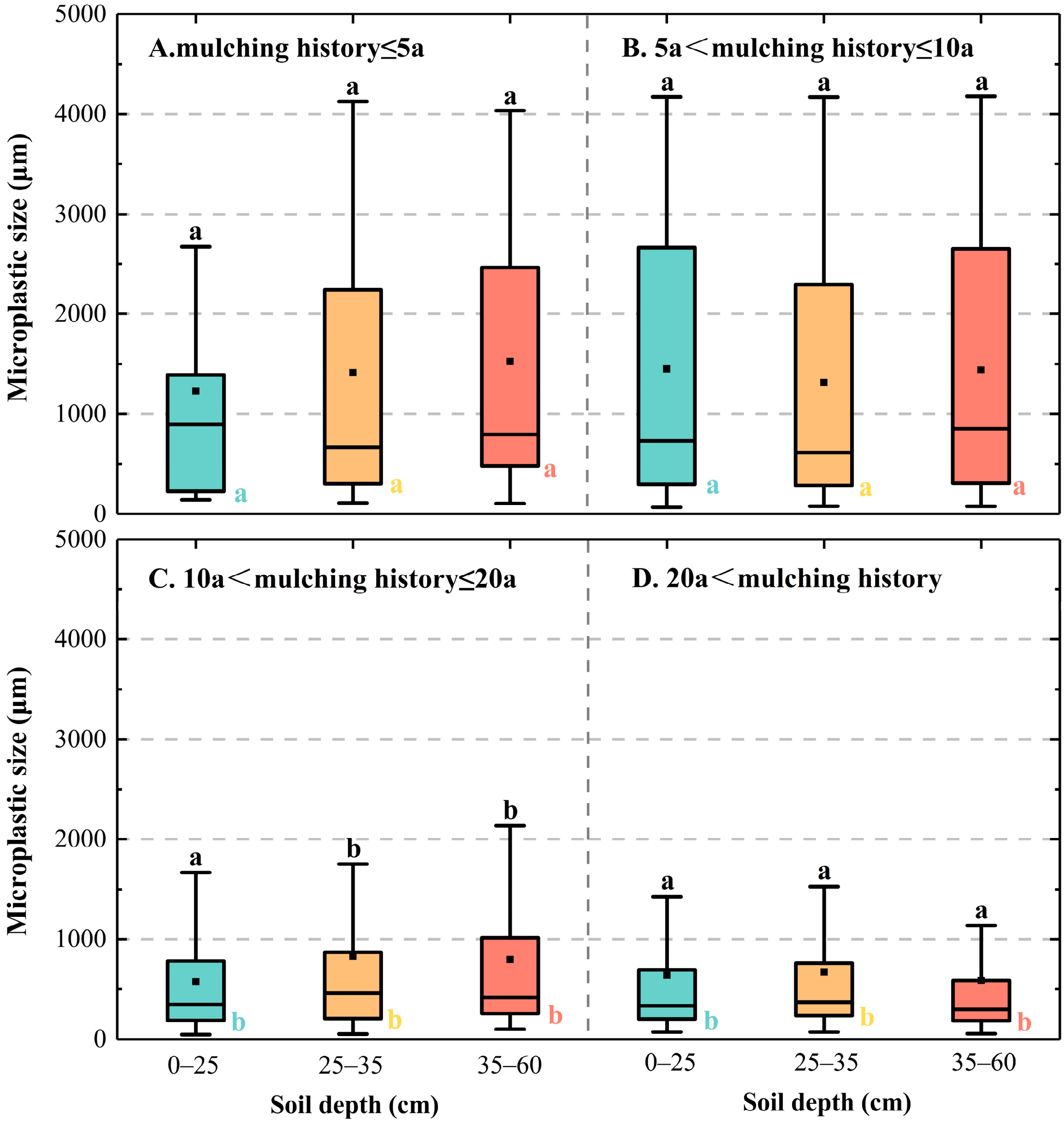

Figure 3. (**A–D**) The size distribution of microplastics in different soil depths with different mulching histories. Difference color columns represent different soil depth. Box boundaries indicate the 25th and 75th percentiles; black line and dot within the box: median and mean, respectively; whiskers below and above the box: minimum and maximum values, respectively. The different lowercase letters in black and color letters show the difference among the different soil depths and mulching history, respectively ($p < 0.05$).

The signs of degradation including cracks and pitting on the surface of film microplastics are shown in Figure 5. It indicated that mulching film residues may be broken down through photooxidation or soil-particle mechanical abrasion [47]. Photooxidation is a process by which the plastic material breaks down due to the effects of sunlight and oxygen. When mulching film is left on the soil surface, exposure to sunlight and oxygen can cause the plastic to break down over time. However, the breakdown process may take a long time, and the results showed that the longer the mulching history, the higher the abundance of microplastics in soils. Due to the large surface area and strong hydrophobicity of microplastics, they can be used as carriers for many organic substances. Therefore, pollutants attached to the surface (such as heavy metals, organic pollutants, etc.) may pose greater risks to the environment. The mulching film commonly used in local cotton fields is transparent, so the microplastics are the same color.

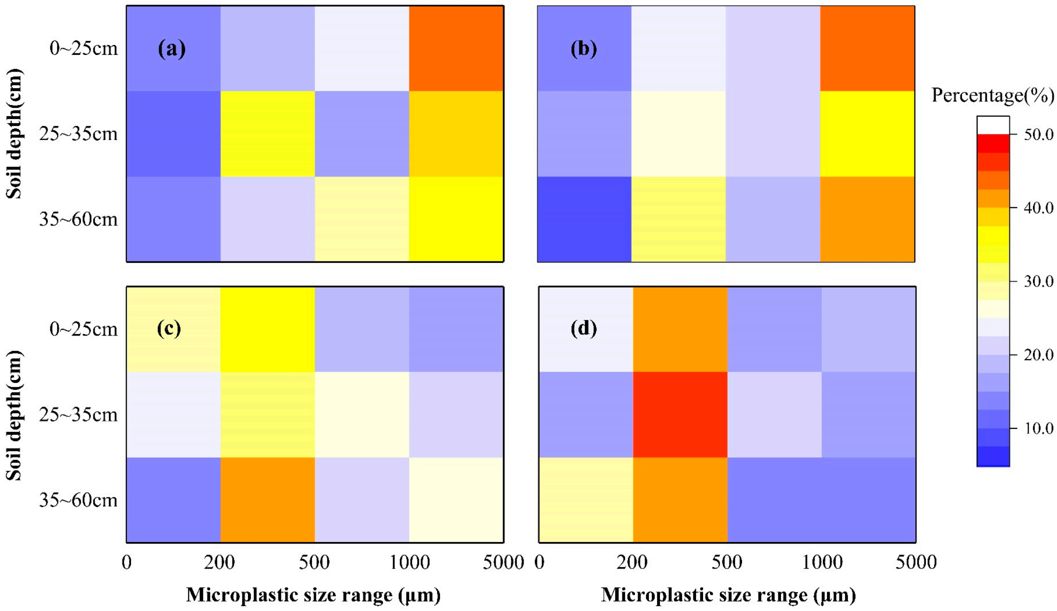

Figure 4. Size distribution of microplastics in different soil depths and mulching history. (**a**) mulching history ≤ 5 years, (**b**) 5 years < mulching history ≤ 10 years, (**c**) 10 years < mulching history ≤ 20 years, (**d**) mulching history > 20 years.

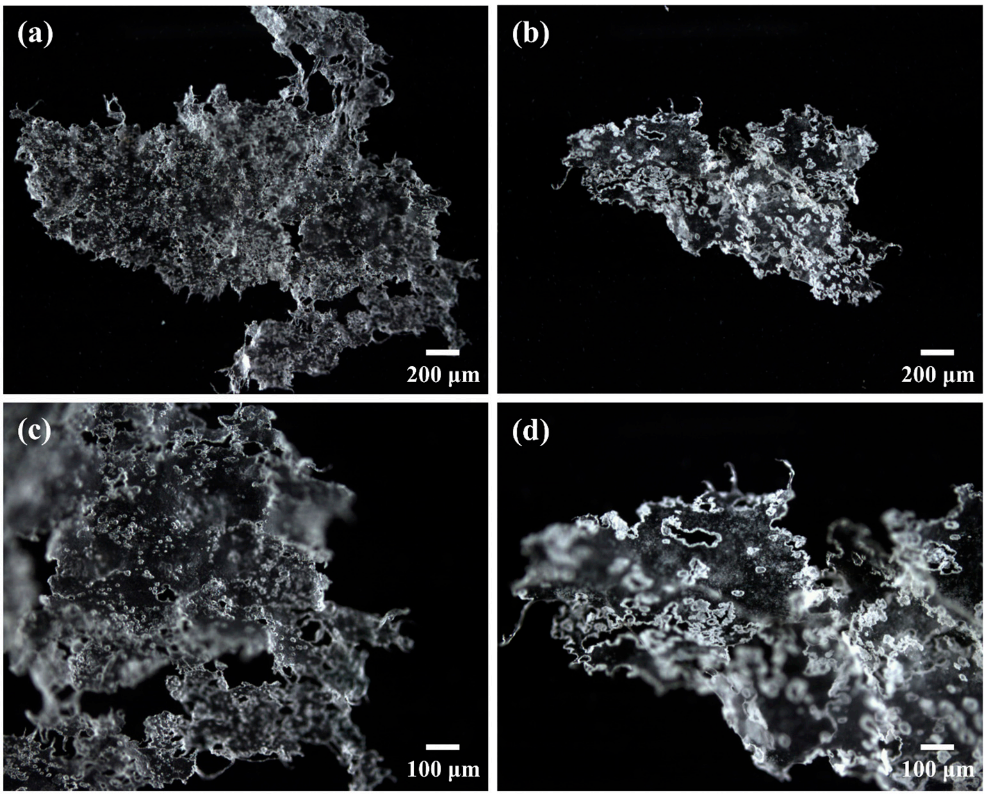

Figure 5. Micrograph images of the film microplastics at 50× (**a**,**b**) and 100× (**c**,**d**) magnifications.

3.3. Relationship between Microplastic Abundance and Mulching History

There are many anthropogenic activities related to agricultural production, but the abundance of microplastics is likely to increase with increasing years of mulching film use. The best polynomial-fitting curve was found between microplastic abundance and mulching history at different soil depth layers as displayed in Figure 6. The coefficients of determination ranged from 0.51 to 0.62. The polynomial fitting curve can quantitatively describe the relationship between the abundance of microplastics and the age of mulching film. Based on these results, the abundance of microplastics at different soil depth layers in any number of years of continuous mulching could easily be calculated. In addition, it appears that the mean abundance of microplastics in the deeper soil layer was lower than that in the surface soil under the same mulching history. Therefore, the mulching history could affect the distribution and accumulation of microplastics in local cotton soils.

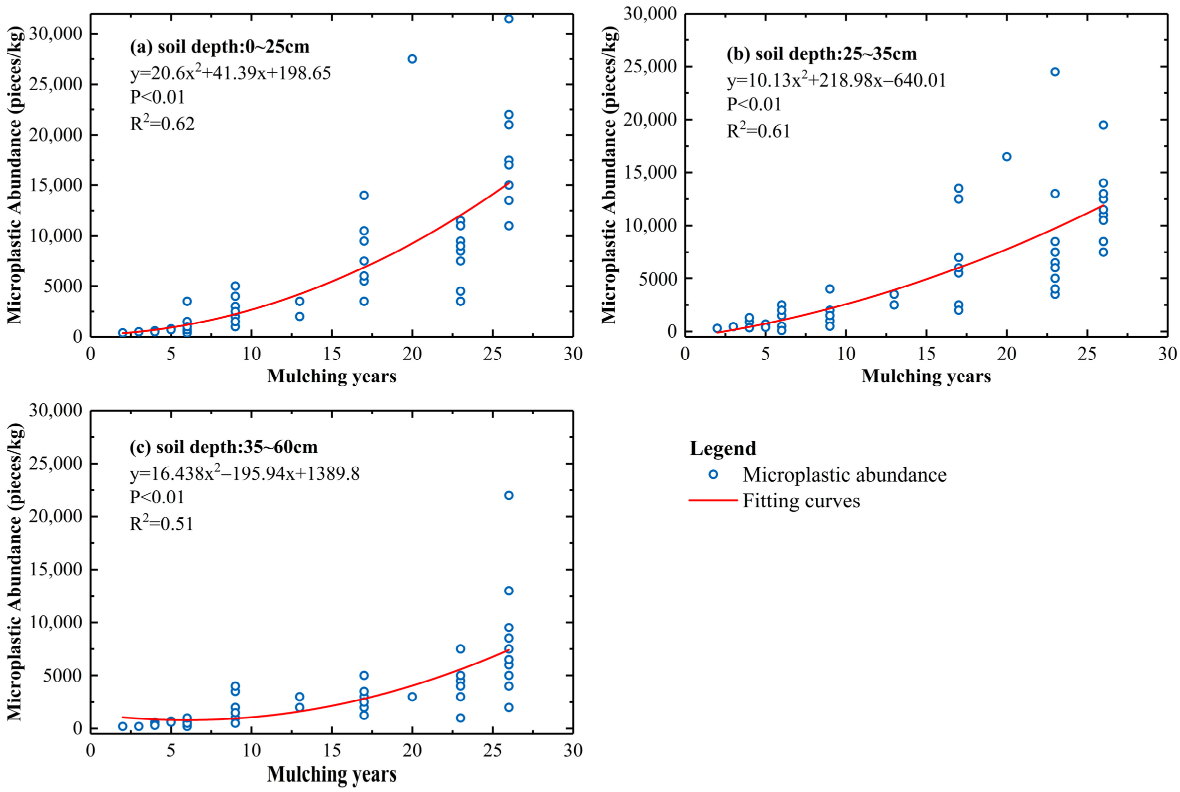

Figure 6. The best polynomial fitting curve between mulching history and microplastic abundance.

4. Discussion

In order to accurately investigate the contamination of microplastics in agricultural soil, this study proposed a reliable method to extract the microplastics from soil. The most common-used and effective method for extracting microplastic particles from soil is based on density separation [48–50]. The principle of this method is due to the density difference between microplastics and environmental matrixes. However, these methods are usually time-consuming, and it is hard to deal with a number of soil samples at the same time. The method of extraction of microplastics that was used in this study can fill these gaps. Furthermore, there are several methods for the identification of microplastics, such as visual inspection methods [51], spectroscopic methods [52] and thermal analysis methods [53]. The study used the visual inspection methods in which microplastics were counted and identified using a microscope. This method used centrifugation technology during microplastic extraction, which can take soil samples in batches with a short period of time [39].

The migration of microplastics from the surface soil to deeper soil layers may be related to multiple factors, such as bioturbation, tillage, water infiltration and so on [34,54]. However, the effects of these external forces on the vertical transport of microplastics were limited. Our results showed that the average abundance of microplastics in cotton fields decreased with soil layer depth. Although there are differences in the magnitude of microplastic abundances, the vertical distribution pattern of microplastic abundances from surface soil layers to deeper soil layers is consistent with other studies [29,37,39].

Some studies indicated that the migration of microplastics into deeper soil may cause more environment issues, including the possible movement of microplastics to groundwater [22].

The abundances of microplastics found in this study were higher than previous studies. For instance, Zhou [7] found that the abundance of microplastics in mulching soils was 571 pieces/kg in eastern China; Yu [29] concluded that agricultural soils of three soil layers had a microplastic abundance of 310 to 5698 pieces/kg in northern China; and Ding [55] reported that the concentration of microplastics in the agricultural soil ranged from 1430 to 3410 pieces/kg in northwestern China. This indicated the high variety of microplastic abundances among the different study areas. The inconsistency of the results in the different studies may be related to many reasons, such as bioturbation, plastic mulching, precipitation, runoff, crop management, crop type, and mechanical disturbance. The differences in the microplastics size in the cotton soil between different years of continuous mulching may be attributed to multiple reasons. The results demonstrated that large-sized microplastics were more likely to accumulate in the field with a short number of years of continuous mulching, while small-sized microplastics were more likely to accumulate in the field with a long number of years of continuous mulching. The large-sized microplastics gradually fragmented into small-sized microplastics, which may be caused by high temperatures, UV radiation and mechanical abrasion, which take a long time [36,56]. The average size of microplastics in deeper soil layers was smaller than in the surface soil under a mulching history of over 10 years. This trend may be because small-sized microplastics are easily leached downwards to deep soil layers through tiny pores and cracks, while large-sized microplastics are likely to be left in the surface soil [51].

Microplastics generally detected in soil and water include five shapes, namely fragments, films, fibers, pellets and foams [15,29]. The multiple input sources of microplastics in agricultural soil have been reported, such as mulching film usage, irrigation, fertilizer, road traffic dust, flooding, human activity and so on [57,58]. Xinjiang is the largest land area in China's provinces and regions, located in northwestern China. However, this region has a typical arid continental climate and a tremendous amount of cotton fields but small population. The drip irrigation method with groundwater was widely used in local cotton fields, and the water source for irrigation was almost without microplastic pollution. Thus, mulching film residues have almost become the only source of microplastics in Xinjiang cotton fields. In this study, film-like microplastics were mainly detected in soil, and other shapes of microplastics were not discussed because they were rare. Mulching film was made by PE, which is the dominant microplastic polymer type in local cotton soils, and similar results have been found in other studies [29,31,55].

The abundance of microplastics in this study shows a significant correlation with mulching history. According to the current survey, Xinjiang cropland presented a greater mulching residue than the national film residue standard, and the plastic mulching history had a significantly positive correlation with residual film [32]. Chen [59] and Yu [29] developed linear regression models to quantitatively describe the impacts of mulching history on microplastic abundance; these studies indicated our results are reliable, but the models vary slightly across regions. Although the local farmers claimed they would recycle plastic mulch films after mechanical harvests, some plastic mulch films would inevitably be broken and remain in the fields. Because the polymer of PE nearly does not biodegrade in natural environments, it would lead to the continuous accumulation of residual film in the soil, which would gradually fragment into microplastics. This study only collected soil samples from cotton fields, but most crops fields had the contribution to the total amounts of residual film in Xinjiang. Therefore, the development of biodegradable films and efficient recovery technologies deserves further study and scrutiny by researchers and policymakers.

5. Conclusions

The abundance and size of microplastics were investigated in Xinjiang cotton fields. The results demonstrated that the surface (0–25 cm) soil layer contained far more microplastics than the middle (25–35 cm) and deep (35–60 cm) soil layers. The microplastics with sizes of 1000–5000 and 200–500 μm were dominant in local cotton fields with under 10 years and over 10 years of continuous mulching history, respectively. The abundance of microplastics increased and the size of microplastics decreased gradually as the years of continuous mulching film increased. The main shape of microplastics in local cotton soils was film, and PE was the prevailing polymer type. The results also clearly showed that plastic mulching film is the dominant source of microplastics in cotton fields, indicating that agricultural soils are the significant environmental reservoirs of microplastics. In addition, the abundance of microplastics in this study shows a significant positive correlation with mulching history. The effects of other factors (e.g., irrigation method, soil texture, leaching process) on the distribution of microplastics deserved to be further explored.

Overall, this study highlights the problem of microplastic distribution in different soil layers in typical arid regions with different mulching histories and provides important data for further research on the risk of microplastics to terrestrial systems under different mulching years. Microplastic pollution-prediction based on mulching history will be studied in future research.

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