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Distribution of microplastics in soil aggregates after film mulching

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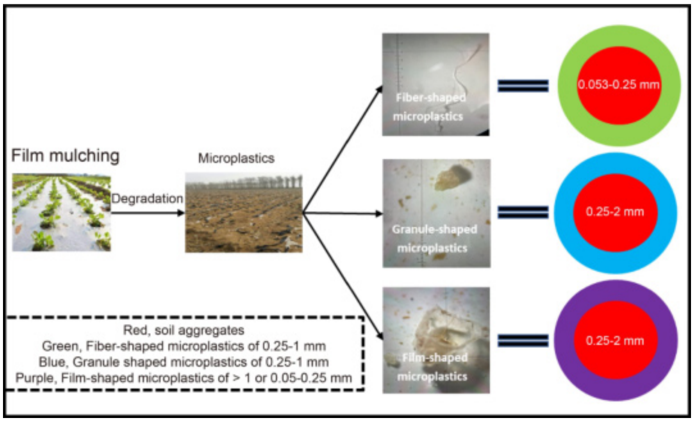
ABSTRACT

- Film mulching decreased soil organic C content in soil aggregates with 0.053–0.25 mm diameter.
- Fiber-shaped microplastics readily combined with the soil aggregates of 0.053–0.25 mm in diameter.
- Film- and granule-shaped microplastics were dominant in 0.25–2 mm soil aggregates.
- Natural and human activities changed the shape and size distribution of particle in soil.

Microplastic distribution is non-homogeneous in agricultural soil following plastic film degradation. However, the distribution of microplastics by shape and particle size in different soil aggregates remains unknown. To elucidate the distribution of microplastic shapes and particle sizes in soil aggregates with increasing years of film mulching, four paired fields with film mulching (FM) and no mulching (NM) were examined at 1, 5, 10, and 20 years after continuous mulching. An increase in soil aggregates of 0.053–0.25 mm diameter was observed; however, soil organic carbon content decreased after long-term FM. Microplastics primarily combined with 0.053–2 mm soil aggregates. Specifically, long-term FM was associated with dominance of film- and fiber-shaped microplastics in soil aggregates of 0.25–2 mm and 0.053–0.25 mm diameter, respectively. Fiber- and granule-shaped microplastics of 0.25–1 mm diameter primarily combined with 0.053–0.25 and 0.25–2 mm soil aggregates, respectively. Film-shaped microplastics of diameter > 1 mm and diameter 0.05–0.25 mm primarily combined with 0.25–2 mm soil aggregates. Therefore, distribution of microplastics in soil aggregates can be used to monitor soil health and quality, greatly enhancing our understanding of the risk posed by microplastics to the environment.



Keywords film mulching, microplastic, soil aggregate, agricultural soil

1 Introduction

In the global agricultural system, approximately 5–35 kg km^{-2} plastic film (of 4–8 μm) is annually used as mulch for a better production in quantity and quality (Liu et al., 2014; Le Moine and Ferry, 2019). Although plastic film mulching supports grain and cash crop yields (Sun et al., 2020), it is also associated with ecological consequences such as soil degradation, groundwater pollution, and surface water eutrophication (Qi et al., 2020). Plastic mulch is difficult to biodegrade, so it is fragmented into microplastic particles (< 5 mm in size) by tillage and UV radiation, and remains in the surface soil (0−20 cm) for long periods due to the low light and oxygen conditions in agricultural soils (Zhou et al., 2020; Xiao et al., 2021). Microplastic accumulation can affect the biophysical properties of the soil (Rudnik and Briassoulis, 2011; De Souza Machado et al., 2019).

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The accumulation of microplastics in agricultural soils likely affects their sustainable development and strongly alters the biogeochemical cycle (Brodhagen et al., 2017; Yu et al., 2021a, 2021b; Shi et al., 2022). For example, microplastics may influence biogeochemical cycles via dissolved organic carbon (DOC) leaching (Wang et al., 2021). Microplastic abundance in soils can exceed 40 000 p kg^{-1} (p, particle) (De Souza Machado et al., 2019), with average levels ranging from 0.34 p kg^{-1} in German agricultural farmland (Piehl et al., 2018) to 160 000 p kg^{-1} in Chinese vegetable fields (Zhou et al., 2019).

Agricultural production practices may indirectly impact polyethylene degradation by affecting soil microorganisms, accelerating the fragmentation of polyethylene particles (Zhang et al., 2020). The resulting microplastic residues affect the physical stability (represented by water molecule bridges) and water binding ability (represented by decreased desorption enthalpy or faster desorption) of soil, and the stability of soil organic matter (SOM) aliphatic crystallites (Fojt et al., 2022; Liu et al., 2022). Aggregates comprise soil particles and organic matter cement together and act as the basic building blocks of soil structure. Aggregate formation and stability are the combined results of physical, chemical, and biological action of soil (Zhang et al., 2019a). The spatial arrangement of primary particles in aggregates forms pore networks, determining the soil structure (Elliott and Coleman, 1988). This in turn controls biogeochemical cycles, soil carbon storage, and carbon processing (Horn et al., 1994), along with the distribution of soil organic carbon (SOC) and other nutrients in soil aggregates (Fojt et al., 2022; Shi et al., 2022a). Yu et al. (2021) reported that microplastic contamination decreased the SOC content in soil aggregates greater than 0.25 mm in diameter, but showed the opposite effect on those < 0.053 mm.

The shapes and sizes of microplastics affect soil aggregation and organic matter decomposition by plant-derived polysaccharides and microbially derived metabolites (Lehmann et al., 2021). The coarse fractions consist of fine fractions plus an organic binder (Wang et al., 2016; Cao et al., 2021), and micron-sized microplastics are bonded to them during the formation of macro-aggregates (Zhang et al., 2022). Usually, microplastics in agricultural soil are classified into three categories: fiber, granule, and film shapes (Li et al., 2022). Fiber-shaped microplastics are rigid and unbranched with thread-like filaments and comprise rayon, polyester terephthalic acid, and polyethylene terephthalate. These microplastics are usually deposited through atmospheric fall out (Li et al., 2022), negatively influencing soil structural stability (Lehmann et al., 2021). Fiber-shaped microplastics are the predominant microplastic type (up to 92%) (Zhang and Liu, 2018). Granule-shaped microplastics are non-spherical, irregular shaped, and mainly composed of poly (N-methylacrylamide) from pesticide formulations, binders in seed coating agents, or flocculants in paper (Exon, 2006). Film-shaped microplastics are planar and rigid shaped, mainly comprising polyethylene from agricultural sources (Huang et al., 2020; Xiao et al., 2021). Different shaped microplastics in the soil matrix bind with different size soil aggregates (Lehmann et al., 2021). Therefore, the distribution of microplastics correlates with soil aggregation, impacting soil fertility, quality, and health (Zhang et al., 2022b). However, few studies have examined the size and shape of microplastics in relation to the particle size classification of soil aggregates in agricultural soil with increasing mulching over the years.

In the present study, we investigated four paired fields with film mulching (FM) and no mulching (NM) in four different years (1, 5, 10, and 20) to elucidate the distribution of microplastic shapes and particle sizes in soil aggregates with increasing years of film mulching. We hypothesized that (1) biological and physicochemical degradation of microplastics would lead to smaller particle size with older mulch age; (2) fiber-shaped microplastics would preferably bind to smaller soil aggregates, whereas granule- and film-shaped microplastics would bind with larger aggregates under long-term FM. This would increase the risk that microplastics pose to the agricultural environment. Understanding the distribution of microplastics in agricultural soil aggregates is relevant to the sustainable development of agricultural practices.

2 Materials and methods

2.1 Study area

The study area was located in Lu Balcony Village, Qin Shi Township, Jiangling County, Jingzhou City, Hubei Province, China (30.15°N, 112.51°E) (Table 1). This area has a subtropical monsoon climate with average annual precipitation of 1000 mm and average annual temperature of 16.2°C. The soil type is fluvo-aquic.

All soil samples were taken in March 2020 after crops were harvested from study plots, and agricultural film was removed. Four sites represented four total durations of continuous mulching: 1, 5, 10, and 20 years (FM plots), with mulching twice per year depending on the crop cultivation (two seasons a year). All film residues were removed after crop harvest. Adjacent triplicate plots without film mulching were selected as controls (NM plots). Three plots were selected as replicates at each site for the four durations of continuous mulching. Soil samples were collected with a coring drill (diameter: 8.5 cm) at a depth of 0–20 cm in the tillage layer. Soil samples were mixed well and refrigerated (−4°C) for subsequent laboratory analysis. For that analysis,

Table 1 Basic information for sampling sites.

	Site 1		Site 2		Site 3		Site 4	
	FM	NM	FM	NM	FM	NM	FM	NM
Duration of continuous mulching (years)	1		5		10		20	
Film amount/year (kg km^{-2})	152	0	456	0	25	0	10	0
Film residues (g m^{-2})	0.691 3		1.302 6		2.021		0.276 8	
Crops	Vegetables	Dry grains	Vegetables and melons	Wheat-rice	Melon-lettuce	Melon-lettuce	Chili-lettuce	Chili-lettuce
Crop yield (kg km^{-2})	52500	13500	45000	7275	22500	22500	26250	26250
Fertilization type	Organic + mineral fertilizer	Mineral fertilizer	Organic + mineral fertilizer	Mineral fertilizer	Mineral fertilizer	Mineral fertilizer	Mineral fertilizer	Mineral fertilizer
Amount of mineral fertilizer (kg km^{-2})	N: 20.8; P: 8; K: 12	N: 26.1; P: 6.4; K: 10.8	N: 24.4; P: 8; K: 9.6	N: 26.1; P: 6.4; K: 10.8	N: 50; P: 5	N: 50; P: 5	N: 50; P: 5	N: 50; P: 5
Amount of organic fertilizer (kg km^{-2})	300	0	240	0	120	0	600	0

NM, no filming; FM, film mulching; N, nitrogen; P, phosphorus; K, potassium.

crop residues were manually removed by sifting through a 5 mm metal mesh. Next, the soil was air-dried at room temperature (24°C) for 30 days and passed through a 2 mm sieve for homogenization. Then SOC, total nitrogen (TN), and total phosphorus (TP) were analyzed as described in section 2.2. Approximately 50 g fresh soil was used from each sample within a week to determine DOC, NH_4^+-N, NO_3^--N, Olsen-P, and microbial biomass C, N, and P (MBC, MBN, and MBP, respectively) levels. Soil samples were also collected from the upper layer (0–20 cm) of each plot based on soil profiles (width 1 m, length 1 m) to determine the amount of mulch residues (g m^{-2}).

2.2 Soil index measurement

SOC and TN content were measured using the potassium dichromate-concentrated sulfuric acid-phenanthroline titration method (Liu et al., 2020). TP was measured using the concentrated sulfuric acid-perchloric acid digestion method (Wei et al., 2002). Soil MBC and MBN were determined using the chloroform fumigation-K_2SO_4 extraction method (Brookes et al., 1985; Vance et al., 1987). C and N concentrations were measured using a Shimadzu TOC-VCPH analyzer (Vwp, Shimadzu, Kyoto, Japan) and a continuous segmented flow analyzer (AA3 HR AutoAnalyzer, SEAL Analytical, USA), respectively. Non-fumigated samples were used to measure DOC, NH_4^+-N, and NO_3^--N according to a published method (Liu et al., 2021). Soil MBP was determined using chloroform fumigation–sodium bicarbonate ($NaHCO_3$) extraction followed by molybdenum antimony colorimetry (Brookes et al., 1982; Wu et al., 2007) using a UV-Vis spectrophotometer (UV-2450, Shimadzu). The non-fumigated samples were used to measure Olsen-P (Olsen et al., 1954; Wei et al., 2019).

2.3 Microplastic extraction and counting

The air-dried soil was sieved and divided into four fractions based on aggregate diameter: > 2, 0.25–2, 0.053–0.25, and < 0.053 mm (John et al., 2005; Dorodnikov et al., 2011). Aggregates were air-dried after wet sieving and weighed to calculate the relative content of each fraction.

Microplastics were extracted from soil using the methods of Li et al. (2022) and Liu et al. (2018), with some modifications. For each size-class sample, 10 g of air-dried soil was transferred to a 250 mL glass beaker, and 100 mL of 6.24 mol L^{-1} $ZnCl_2$ with a density of 1.6 g cm^{-3} was added. The beaker was then sonicated at 60 Hz for 20 min using an ultrasonic cleaning machine (SB-800 DTD, SCIENTZ, China) to break up soil aggregates, and then samples were stirred for 10 min. Samples were left to stand for 24 h to allow separation (low-density polyethylene has a density of 0.91–0.94 g cm^{-3} and floated to the surface). The supernatant was passed through a blended cellulose membrane filter (0.45 μm, Solarbio, Beijing, China). The collected materials were transferred to 100 mL of 30% H_2O_2 solution for 24 h and then heated on a hotplate at 60°C to remove SOC (Wang et al., 2018). Next, the solution was again filtered through a membrane filter (0.45 μm), transferred to a 60 mm diameter Petri dish, and dried at 60°C for 48 h (Liu et al., 2018; Weithmann et al., 2018).

The microplastics on the filter membrane were visually examined using a Stemi 2000-C stereo inspection microscope (Ziess, Oberkochen, Germany) with 50× magnification. Ten fields of view in the areas of the samples surface were randomly selected for each membrane under the microscope. For each field view, the shape, size, color, and quantity of microplastic fragments were recorded. Fibers, granules, and films were classified using the method of Li et al. (2022). Fiber-shaped microplastics were defined as glossy, curled filaments; granular microplastics were defined as glossy, roughly spherical particles; and film-shaped microplastics were generally shiny under light, with rounded edges. The size of microplastics was recorded as the length of the longest side.

2.4 Statistical analysis

Statistical analyses were performed in R software (4.0.0). After the Levene's test, two-way analysis of variance was performed to test the effects of year of continuous mulching, treatment (FM and NM), and treatment–year interactions using the *aov* function (Wei et al., 2019; Liu et al., 2021). Mean values for each year (1, 5, 10, or 20) were compared using the least significant difference at the 5% level ($LSD_{0.05}$) with the "agricolae" package (Wei et al., 2019; Liu et al., 2021).

3 Results

3.1 Soil aggregates

No significant change for all durations was observed in the abundance of soil aggregates < 0.053 mm between FM and NM ($P > 0.05$). Soil aggregates measuring 0.053–0.25 mm were less abundant in FM soil than in NM soil at 1 year ($P < 0.05$), but more abundant in FM soil than in NM soil after 20 years ($P < 0.05$). In FM soil, soil aggregates of this size were more abundant 20 years after continuous mulching than for all other durations ($P < 0.05$). No significant change was observed in the abundance of soil aggregates measuring 0.25–2 mm between FM and NM ($P > 0.05$). Soil aggregates > 2 mm were more abundant in FM soil than in NM soil at 5 years ($P < 0.05$) (Table 2). Overall, FM soil exhibited a greater abundance of aggregates measuring 0.053–0.25 mm than NM soil over 20 years of continuous mulching.

3.2 SOC, TN, and TP

The SOC content in soil aggregates measuring 0.25–2 mm was higher in NM soil than in FM soil for the 5 years samples ($P < 0.05$), but the SOC content was lower in NM soil than in FM soil for the 10 years samples ($P < 0.05$) (Fig. 1A). The SOC content in soil aggregates measuring 0.053–0.25 mm was higher in NM soil than in FM soil for the 20 years samples ($P < 0.05$). For FM soil, the SOC content in soil aggregates of this size was only lower compared with that for the 1 year samples ($P < 0.05$) (Fig. 1B). The SOC content in soil aggregates < 0.053 mm was lower in NM soil than in FM soil at 10 years ($P < 0.05$). For FM soil, the SOC content in soil aggregates < 0.053 mm was higher at 10 years than at 1 year ($P < 0.05$) (Fig. 1C).

The TN content in 0.25–2 mm soil aggregates was higher in NM soil than in FM soil for 5 years ($P < 0.05$), but lower in NM soil than in FM soil after 10 years ($P < 0.05$) (Fig. 1D). In 0.053–0.25 mm aggregates, TN was higher in NM soil than in FM soil after 10 years ($P < 0.05$). For FM soil, the TN content in soil aggregates of 0.053–0.25 mm was higher after 5 years than after 1 year ($P < 0.05$) (Fig. 1E). The TN content in soil aggregates < 0.053 mm exhibited no changes over time in NM or FM soil (Fig. 1F).

The TP content in 0.25–2 mm soil aggregates was higher in NM soil than in FM soil at the 5-year mark ($P < 0.05$). For FM soil, the TP content in 0.25–2 mm aggregates was higher after 10 and 20 years of continuous mulching than after 1 and 5 years ($P < 0.05$) (Fig. 1G). The TP content in 0.053–0.25 mm soil aggregates was higher in NM soil than in FM soil at 5 years ($P < 0.05$). For FM soil, the TP content in 0.053–0.25 mm aggregates was higher after 10 and 20 years than after 1 and 5 years ($P < 0.05$) (Fig. 1H). The TP content in soil aggregates < 0.053 mm was higher in NM soil than in FM soil at 5 years ($P < 0.05$). For FM soil, the TP content in aggregates < 0.053 mm was higher after 10 and 20 years than after 1 and 5 years ($P < 0.05$) (Fig. 1I). Overall, FM was associated with a long-term decrease in SOC content in 0.053–0.25 mm soil aggregates when compared with levels in NM soil but did not appear to affect TN or TP content in aggregates of any size.

3.3 Soil available nutrients and microbial biomass

The DOC content was consistently lower in NM soil than in FM soil across all durations ($P < 0.05$). For FM soil, DOC content was lower after 5 years ($P < 0.05$) but higher after 10 and 20 years ($P < 0.05$) than at 1 year (Fig. S1a). Soil NH_4^+-N content was higher in NM soil than in FM soil at 5 years ($P < 0.05$). For FM soil, the NH_4^+-N content was lower at 5 years than at 1 year ($P < 0.05$) (Fig. S1b). The NO_3^--N content was consistently lower in NM soil than in FM soil at 1 and 5 years ($P < 0.05$); the opposite trend was observed for at 10 and 20 years. For FM soil, the NO_3^--N content was higher after 5 and 10 years than at 1 year ($P < 0.05$) (Fig. S1c). The Olsen-P content was consistently lower in NM soil than in FM soil after 10 and 20 years ($P < 0.05$). For FM soil, the Olsen-P was higher at 5, 10, and 20 years than at 1 year ($P < 0.05$) (Fig. S1d). Overall, FM was associated with

Table 2 Results of two-way ANOVAs showing the effects of year, treatment, and year–treatment interactions on particle size of soil aggregates.

Year	Treatment	Soil aggregate size (mm)			
		<0.53	0.53−0.25	0.25−2	>2
1	NM	23.67 ± 16.25aA	29.18 ± 5.46aA	22.54 ± 13.69bA	2.09 ± 1.73bA
	FM	13.86 ± 11.49αA	10.99 ± 3.72βB	50.52 ± 12.93αA	0.58 ± 0.40αA
5	NM	11.72 ± 1.02abA	23.34 ± 3.73aA	39.51 ± 4.08aA	1.95 ± 0.19bA
	FM	20.13 ± 10.62αA	18.81 ± 2.97αβA	37.16 ± 12.54αA	0.58 ± 0.36αB
10	NM	13.99 ± 5.39abA	14.11 ± 2.77bA	46.51 ± 6.86aA	2.72 ± 1.35bA
	FM	19.66 ± 6.65αA	14.66 ± 3.68βA	42.46 ± 5.69αA	0.56 ± 0.33αA
20	NM	7.36 ± 1.71bA	8.11 ± 1.85bB	53.97 ± 7.37aA	7.97 ± 4.93aA
	FM	17.15 ± 11.33αA	28.16 ± 11.55αA	31.24 ± 22.59αA	0.53 ± 0.27αA

	Soil aggregate size (mm)							
	<0.53		0.53−0.25		0.25−2		>2	
Factor (df)	*F*	*P*	*F*	*P*	*F*	*P*	*F*	*P*
Year (3)	0.501	0.687	1.866	0.176	0.557	0.651	3.297	*
Treatment (1)	0.831	0.376	0.06	0.809	0.003	0.9543	15.763	**
Years × Treatment (3)	1.374	0.287	13.409	***	4.509	*	3.408	*

Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatment in NM and FM, respectively. Latin uppercase letters represent significant differences between NM and FM at $P < 0.05$. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P < 0.05$, < 0.01, and < 0.001, respectively. All results are means ± standard deviation ($n = 3$). NM, no mulching; FM, plastic film mulching.

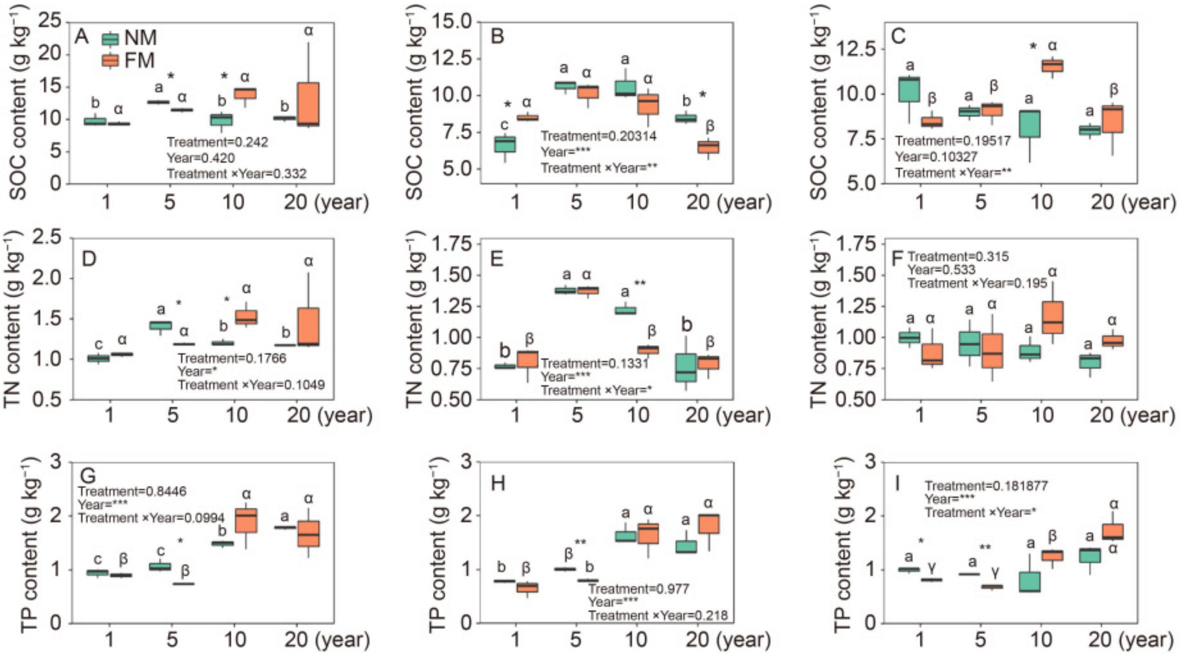

Fig. 1 Changes of soil organic carbon, total nitrogen, and total phosphorus over time in soils with (FM) or without (NM) plastic film mulching in soil aggregates measuring 0.25–2 (A, D, G), 0.053–0.25 (B, E, H), and < 0.053 (C, F, I) mm. SOC, soil organic carbon; TN, total nitrogen; TP, total phosphorus. Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatments in NM and FM, respectively. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P < 0.05$, < 0.01, and < 0.001, respectively. $n = 3$.

long-term increases in soil DOC, NO_3^--N, and Olsen-P content when compared to NM.

No significant difference was observed in MBC, MBN, and MBP between FM and NM soils ($P > 0.05$), expect for MBN and MBP at 10 and 5 years, respectively. In FM soil, the MBN and MBP content increased over time ($P < 0.05$); FM appeared to have no long-term influence on MBN, MBC, or MBP (Fig. S2a–c).

3.4 Total microplastic content

No significant difference was observed in total microplastic content in 0.25–2 mm aggregates between FM and NM soils ($P > 0.05$). For NM soil, total microplastic content in 0.25–2 mm aggregates was higher after 10 and 20 years than at 1 year ($P < 0.05$); for FM soil, the highest total microplastic content in this aggregate size class was observed after 20 years (Fig. 2A). Total microplastic content in 0.053–0.25 mm soil aggregates was higher in NM soil than in FM soil at 5 years ($P < 0.05$), but was lower in NM soil than in FM soil at 10 years ($P < 0.05$). Total microplastic content in 0.053–0.25 mm soil aggregates was higher after 10 and 20 years than at 1 and 5 years in both NM and FM soils ($P < 0.05$) (Fig. 2B). No significant difference was observed in total microplastic content in soil aggregates < 0.053 mm between FM and NM soils ($P > 0.05$). For NM soil, total microplastic content in soil aggregates < 0.053 mm showed no significant change with time ($P > 0.05$), but total levels increased over 20 years in FM soil aggregates of this size (Fig. 2C). Overall, total microplastic content in soil aggregates fluctuated over time, but FM increased total microplastic content in soil aggregates measuring 0.25–2, 0.053–0.25, and < 0.053 mm over time.

3.5 Microplastic shape

In 0.25–2 mm soil aggregates, no significant changes were observed in the abundance of fiber-shaped microplastics over time in NM or FM soil ($P > 0.05$) (Fig. 3A). Granule-shaped microplastic content was higher in NM than in FM soil at 10 years ($P < 0.05$). In FM soil, granule-shaped microplastics were more abundant at 5 years than at 1 year ($P < 0.05$) (Fig. 3B). The film-shaped microplastic content was lower in NM than in FM soil after 20 years ($P < 0.05$). For FM soil, film-shaped microplastics were more abundant at 10 and 20 years than at 1 and 5 years ($P < 0.05$) (Fig. 3C).

In 0.053–0.25 mm soil aggregates, the fiber-shaped microplastic content was higher in NM soil than in FM soil at 10 and 20 years ($P < 0.05$). For FM soil, fiber-shaped microplastics were more abundant at 10 and 20 years than at 1 and 5 years ($P < 0.05$) (Fig. 3D). Granule-shaped microplastics were more abundant in NM than in FM at 5 years ($P < 0.05$), but less abundant in NM than in FM at 10 years ($P < 0.05$). For FM soil, this microplastic shape was more abundant at 10 and 20 years than at 1 and 5 years ($P < 0.05$) (Fig. 3E). Levels of film-shaped microplastics were higher in NM than in FM soil at 10 years ($P < 0.05$). In FM soil, film-shaped microplastics were more abundant at 10 years than at all other time points ($P < 0.05$) (Fig. 3F).

In soil aggregates < 0.053 mm, fiber-shaped microplastics were more abundant in NM than in FM soil at 10 years ($P < 0.05$). In FM soil, fiber-shaped microplastics were more abundant at 10 and 20 years than at 1 and 5 years ($P < 0.05$) (Fig. 3G). In FM soil, the content of granule-shaped microplastics showed no significant changes with time ($P > 0.05$) (Fig. 3H). Film-shaped microplastics were less abundant in NM than in FM soil at 1 year ($P < 0.05$). In FM soil, levels of film-shaped microplastics were higher after 10 and 20 years of film mulching than after 1 and 5 years ($P < 0.05$) (Fig. 3I). Overall, long-term FM was associated with increased abundance of film- and fiber-shaped microplastics in soil aggregates measuring 0.25–2 and 0.053–0.25 mm, respectively.

3.6 Size distribution of fiber-, granule-, and film-shaped microplastics

The abundance of fiber-shaped microplastics > 1, 0.25–1, and 0.05–0.25 mm in soil aggregates measuring 0.25–2 mm

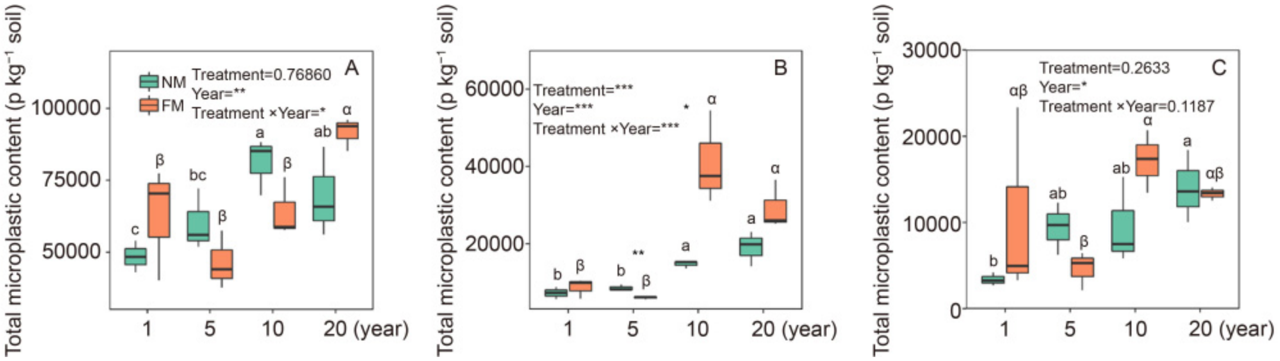

Fig. 2 Changes in total microplastic shapes over time in soils with (FM) or without (NM) plastic film mulching in soil aggregates measuring 0.25–2 (A), 0.053–0.25 (B), and < 0.053 (C) mm. Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatments in NM and FM, respectively. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P < 0.05$, < 0.01, and < 0.001, respectively. $n = 3$. p, particle.

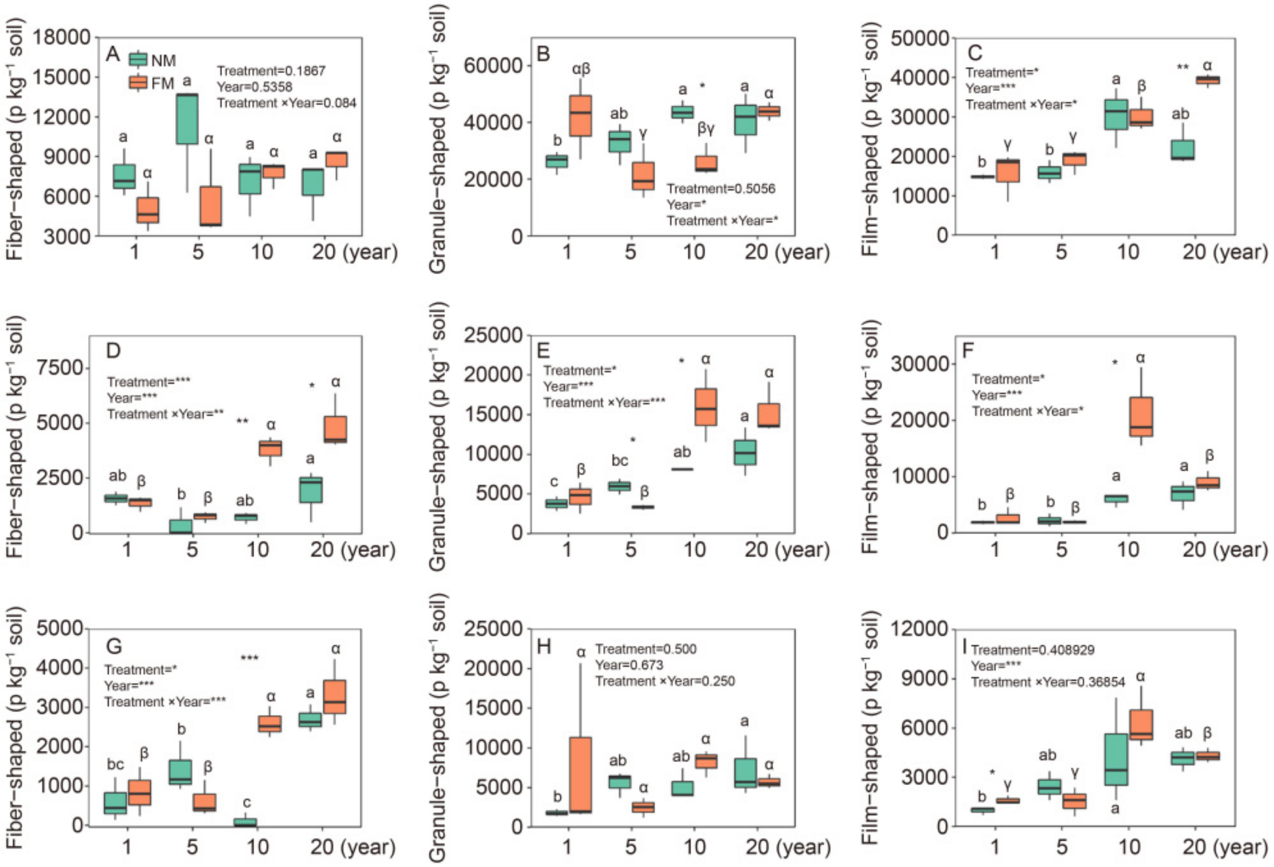

Fig. 3 Changes in abundance of three different microplastic shapes (fiber-shaped, granule-shaped, and film-shaped) over time in soils with (FM) or without (NM) plastic film mulching in soil aggregates measuring 0.25–2 (A–C), 0.053–0.25 (D–F), and < 0.053 (G–I) mm. Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatments in NM and FM, respectively. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P < 0.05$, < 0.01, and < 0.001, respectively. $n = 3$. p, particle.

was not significantly different over time for either NM or FM soils ($P > 0.05$) (Fig. 4A–C), and fiber-shaped microplastics > 1 mm in soil aggregates measuring 0.053–0.25 mm was also not different between NM and FM soils (Fig. 4D). Fiber-shaped microplastics measuring 0.25–1 mm in soil aggregates measuring 0.053–0.25 mm were less abundant in NM than FM soil at 10 and 20 years ($P < 0.05$) (Fig. 4E). Fiber-shaped microplastics of 0.05–0.25 mm in 0.053–0.25 mm soil aggregates were less abundant in NM than in FM soil at 10 years ($P < 0.05$) (Fig. 4F). Fiber-shaped microplastics > 1 mm in aggregates < 0.053 mm were less abundant in NM than in FM soil at 10 years ($P < 0.05$) (Fig. 4G). Fiber-shaped microplastics measuring 0.25–1 mm in soil aggregates < 0.053 mm were less abundant in NM than FM soil at 10 years ($P < 0.05$) (Fig. 4H). Fiber-shaped microplastics measuring 0.05–0.25 mm in soil aggregates showed no significant change over time between NM and FM soils ($P > 0.05$) (Fig. 4I). Overall, FM treatment was associated with high levels of 0.25–1 mm fiber-shaped microplastics in soil aggregates measuring 0.053–0.25 mm.

Granule-shaped microplastics measuring 0.25–1 mm in aggregates measuring 0.25–2 mm were less abundant in NM than in FM soil at 20 years ($P < 0.05$) (Fig. 5A). Granule-shaped microplastics measuring 0.05–0.25 mm in soil aggregates measuring 0.25–2 mm were more abundant in NM than in FM soil at 10 years ($P < 0.05$) (Fig. 5B). There was no significant difference in granule-shaped microplastics measuring 0.25–1 mm in aggregates measuring 0.053–0.25 mm between FM and NM soils ($P > 0.05$) (Fig. 5C). Granule-shaped microplastics measuring 0.05–0.25 mm in soil aggregates measuring 0.053–0.25 mm were more abundant in NM than in FM soil at 5 years ($P < 0.05$), but were less abundant in NM than in FM soil at 10 years ($P < 0.05$) (Fig. 5D). There was no significant difference in granule-shaped microplastics measuring 0.25–1 mm in aggregates measuring < 0.053 mm between FM and NM soils (Fig. 5E). Granule-shaped microplastics measuring 0.05–0.25 mm in soil aggregates < 0.053 mm did not significantly change with film age between NM and FM soils (Fig. 5F). Overall, FM treatment was associated with high abundance of 0.25–1 mm granule-shaped microplastics in soil aggregates measuring 0.25–2 mm.

Film-shaped microplastics > 1 mm in soil aggregates measuring 0.25–2 mm were more abundant in NM than in

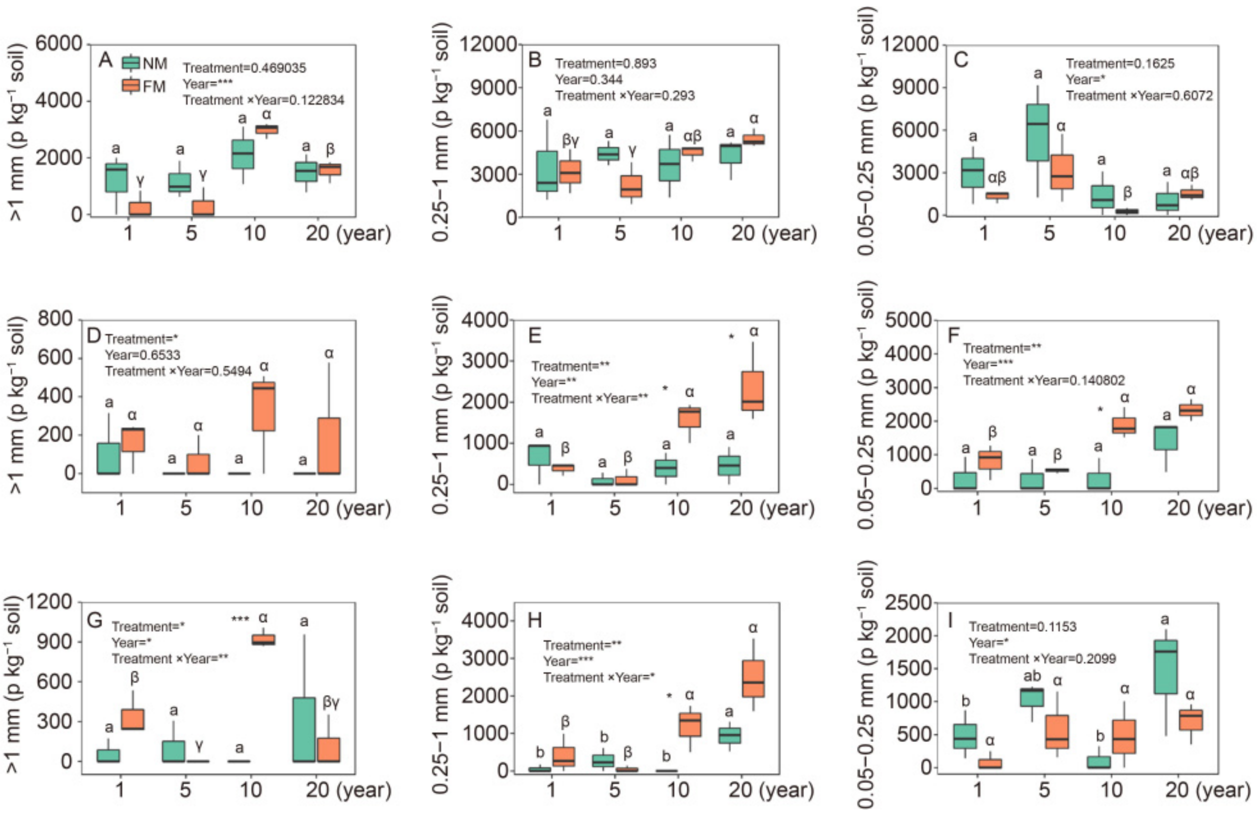

Fig. 4 Abundance of three different microplastics sizes (> 1, 0.25–1, and 0.05–0.25 mm) of fiber-shaped microplastic over time in soils with (FM) or without (NM) plastic film mulching in three soil aggregate particle sizes (0.25–2 (A–C), 0.053–0.25 (D–F), and < 0.053 (G–I) mm). Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatments in NM and FM, respectively. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P <$ 0.05, < 0.01, and < 0.001, respectively. $n = 3$. p, particle.

FM soil at 10 and 20 years ($P < 0.05$) (Fig. 6A). Film-shaped microplastics measuring 0.25–1 mm in 0.25–2 mm soil aggregates were more abundant in NM than FM soil at 1 year ($P < 0.05$) (Fig. 6B). Film-shaped microplastics measuring 0.05–0.25 mm in 0.25–2 mm soil aggregates were more abundant in NM than in FM soil after 10 years ($P < 0.05$), but were less abundant in NM than in FM soil after 20 years ($P < 0.05$) (Fig. 6C). Film-shaped microplastics > 1 mm were not found in 0.053–0.25 mm aggregates of either soil type (Fig. 6D). Film-shaped microplastics measuring 0.25–1 mm in 0.053–0.25 aggregates were less abundant in NM than in FM soil after 10 years ($P < 0.05$) (Fig. 6E). Film-shaped microplastics in 0.053–0.25 mm soil aggregates did not significantly differ between NM and FM ($P > 0.05$) (Fig. 6F). For FM and NM soils, 0.25–1 mm film-shaped microplastics in aggregates < 0.053 mm did not significantly change over time ($P > 0.05$) (Fig. 6G). Film-shaped microplastics of 0.05–0.25 mm in aggregates < 0.053 mm did not significantly change over time between NM and FM soils ($P > 0.05$) (Fig. 6H). Overall, long-term FM treatment was associated with increased levels of film-shaped microplastics measuring 0.05–0.25 mm and > 1 mm in 0.25–2 mm soil aggregates.

4 Discussion

Microplastics of different shapes and sizes bind to soil aggregates of different sizes, resulting in uneven distribution of microplastics among soil aggregates.

4.1 Soil aggregates

Microplastics are known to affect soil aggregation in an SOM-dependent manner (Liang et al., 2021; Zhang et al., 2022a, 2022b). In our results, plastic film mulching was associated with an increase in the abundance of soil aggregates measuring 0.053–0.25 mm over 20 years of continuous mulching when compared with NM soil. This is consistent with the results of Wang et al. (2017) who reported that continuous plastic film mulching increased soil aggregate abundance in maize cropping systems. This may have occurred because mulching increased crop root biomass in the upper 20 cm soil layer, enhancing crop roots (plant-derived polysaccharides) and microbial activities (microbially derived metabolites, i.e., polysaccharides and proteins) to increase aggregates via physical, chemical, and biological alterations and helping to enmesh and realign the soil

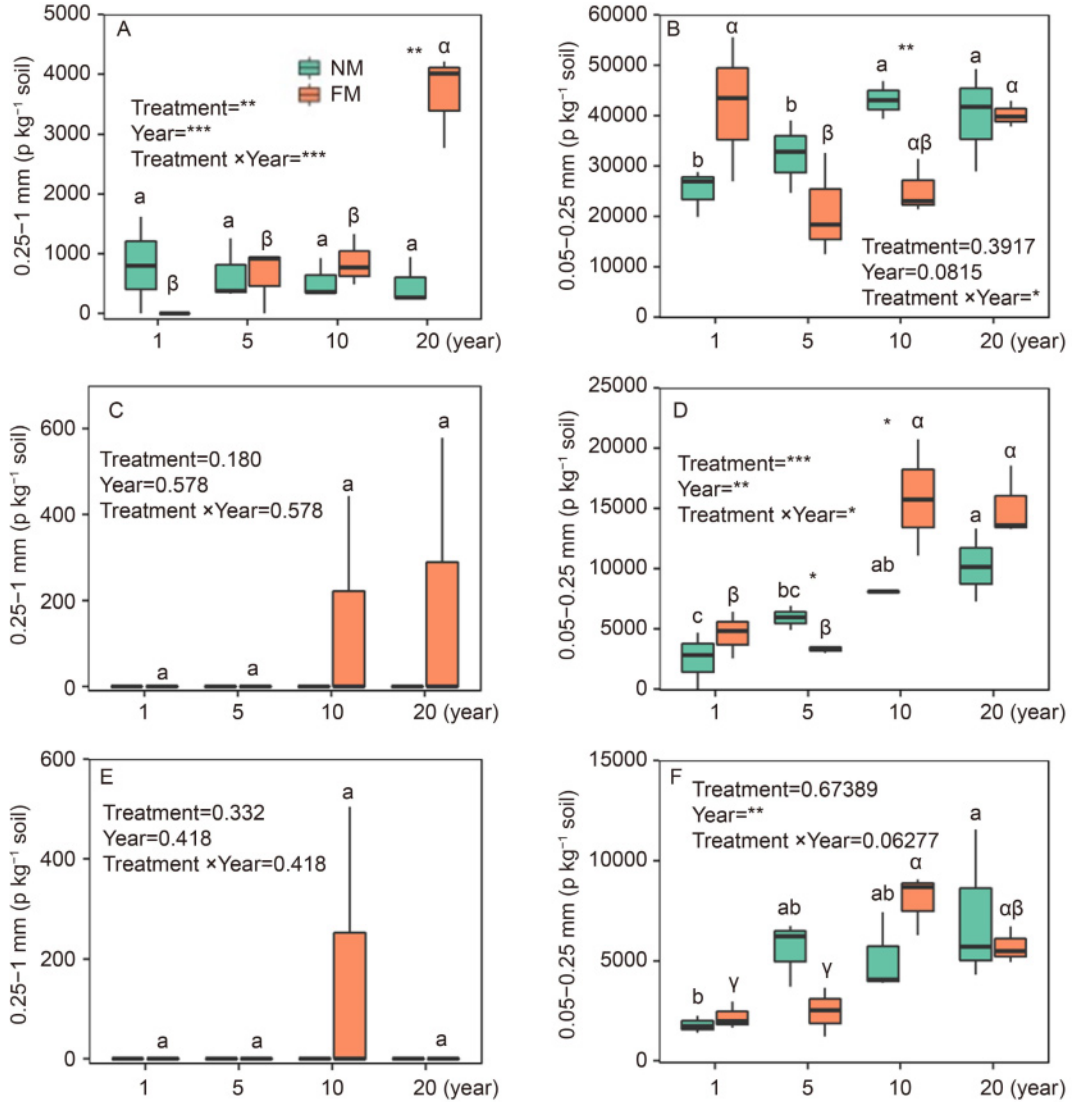

Fig. 5 Abundance of three different microplastics size (0.25–1 and 0.05–0.25 mm) of granule-shaped microplastic over time in soils with (FM) or without (NM) plastic film mulching in three soil aggregate particle sizes (0.25–2 (A, B), 0.053–0.25 (C, D), and < 0.053 (E, F) mm). Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatments in NM and FM, respectively. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P < 0.05$, < 0.01, and < 0.001, respectively. $n = 3$. p, particle.

particles (Haynes and Francis, 1993; Tisdall, 1994; Xue and Wang, 2020). In particular, crop roots and root exudates can alter soil biogeochemical properties and help enmesh and align soil particles, leading to aggregate formation. In addition, as crop residues and microplastics accumulate in soil during years of plastic mulching, microplastics are incorporated into aggregates by microorganisms (Forster, 1990; Catt, 2001; Zang et al., 2020), yielding newly formed aggregates measuring 0.053–0.25 mm and containing microplastics (Zhang et al., 2019a).

4.2 SOM

Crop root growth causes root exudate secretion into the soil, resulting in higher levels of DOC in mulched soil than those in non-mulched soil (Fig. S1) and promoting the downward migration of microplastics along the fissure channels (Ding et al., 2019; Zhu et al., 2019; Guo et al., 2020; Liu et al., 2021; Shi et al., 2022). Furthermore, exudate secretion promotes the entanglement of plastic film with roots, impeding the removal of plastic after harvest (Li et al., 2022). Plastic mulching improves crop yield (Sun et al., 2020), enhancing the return of biomass, in the form of crop residues, to the soil (Zhang et al., 2017). The high biomass in mulched soils was a result of the high SOC levels with low depletion (Zhang et al., 2017). When microbial nutrient limitation is alleviated, the availability of N and P (NO_3^--N and Olsen-P; Fig. S1) increases as related to C availability after fertilization

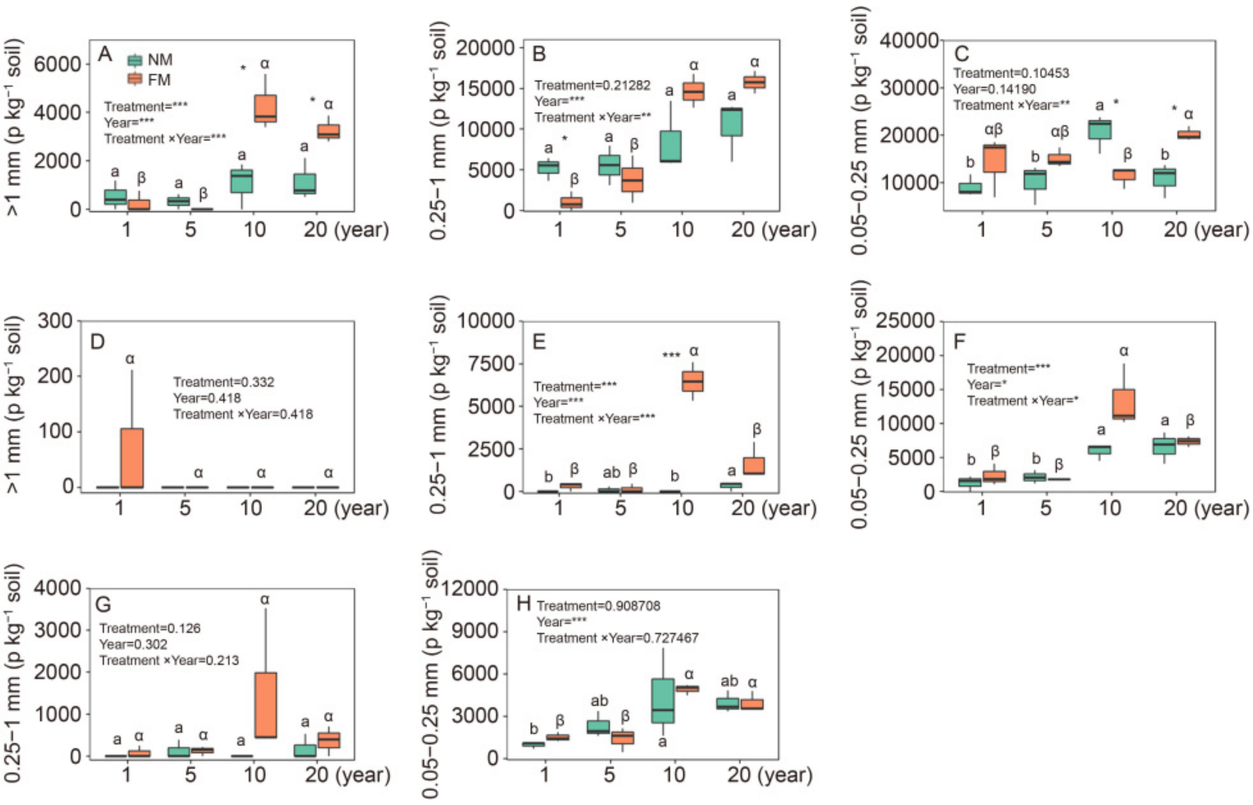

Fig. 6 Abundance of three different microplastics sizes (> 1, 0.25–1, and 0.05–0.25 mm) of film-shaped microplastic over time in soils with (FM) or without (NM) plastic film mulching in three soil aggregate particle sizes (0.25–2 (A–C), 0.053–0.25 (D–F), and < 0.053 (G, H) mm). Different Latin and Greek lowercase letters indicate significant differences ($P < 0.05$) between treatments in NM and FM, respectively. *, **, and *** represent significant effects of year, treatment, or year–treatment interaction at $P <$ 0.05, < 0.01, and < 0.001, respectively. $n = 3$. p, particle.

(Schimel and Weintraub, 2003; Ludewig et al., 2019; Wang et al., 2019). Our results showed that FM soil aggregates measuring 0.053–0.25 mm exhibited lower SOC content than comparable NM aggregates over time. This may have occurred because mulching increases soil temperature and water-use efficiency (Adams et al., 1995; Kirschbaum et al., 2001; Wiesmeier et al., 2015). Therefore, according to our results, long-term FM preferentially caused SOC mineralization in 0.053–0.25 mm soil aggregates (Cuello et al., 2015; Lee et al., 2019). Usually, SOM (root exudates and crop residues) promotes stable soil aggregation by stimulating microbial growth and metabolism, promoting plant and microbial metabolite (polysaccharides and proteins) production and facilitating aggregate stabilization (Caesar-Tonthat, 2002; Tisdall, 1994). However, due to the potential toxicity of film residues, the desorption of additives and their transition to the soil can affect soil processes such as microbial activity, subsequently affecting soil aggregation by altering metabolite levels (Caesar-Tonthat, 2002; Judith et al., 2012). After some durations of continuous mulching, the SOC, TN, and TP levels were higher in NM soil aggregates than in FM soil aggregates or were similar between soil types, indicating that the effects of organic matter on aggregation varied due to different crops (Abiven et al., 2009; Liang et al., 2021). Furthermore, we found that long-term FM did not affect MBC, MBN, or MBP, although differences were observed between NM and FM soil after some durations of continuous mulching (Fig. S2). This suggests that microplastics-based carbon is relatively stable and not readily available to microorganisms (Steinmetz et al., 2016; Yu et al., 2021a; Tong et al., 2022).

4.3 Distribution of microplastic shapes and sizes

Soil fertilization increases the biodiversity and abundance of several genera of soil microorganisms capable of degrading low-density plastic film, thereby accelerating the fragmentation of plastic film particles (Zhang et al., 2020). The distribution of microplastic shapes varied with soil aggregate size under FM conditions in our study. The size and shape of microplastic fragments are diverse and vary along continuous scales (Kooi and Koelmans, 2019). Microplastics comprise polymers and chemical additives at various states of weathering, and environmental conditions can enhance their complexity, for example through absorption of chemical contaminants and biofilm formation (Galloway et al., 2017; Burns and Boxall, 2018). Our results showed that FM treatment was associated with high levels of film-shaped microplastics in 0.25–2 mm soil aggregates. In addition, we found that most film-shaped microplastics were > 1 mm and favorably bound with 0.25–2 mm soil aggregates. With increasing film age, film-shaped microplastics degrade into smaller fragments (Yu et al., 2021a, 2021b; Shi et al., 2022), explaining why the 0.05–0.25 mm film-shaped microplastics were more abundant after 20 years than after 1 year.

Fiber-shaped microplastics are composed of rayon, polyester terephthalic acid, and poly (ethylene terephthalate), and these very likely originate from textiles (Zhang et al., 2019b; Li et al., 2022) or from weathered plastic film debris and string routinely used for vegetable cultivation in our study area (Zhou et al., 2019; Kim et al., 2021). Fiber-shaped microplastics measuring 0.25–1 mm in 0.053–0.25 mm soil aggregates became more abundant in FM than in NM soil over time, supporting the idea that these microplastics can induce fracture points in newly formed aggregates, facilitating breakdown under physical disturbance (Zhang and Liu, 2018; Rillig et al., 2019). Curliness, flexibility, and surface properties of fiber-shaped microplastics can potentially affect aggregate formation (Lehmann et al., 2021). In our study, fiber-shaped microplastics measuring 0.25–1 mm were bonded to 0.053–0.25 mm soil aggregates during the formation of macro-aggregates (Wang et al., 2016; Cao et al., 2021; Zhang et al., 2022a).

Granule-shaped microplastics showed similar abundance in FM and NM soils, suggesting that these particles do not easily bind to soil aggregates (Zhang and Liu, 2018). This result contradicts that of Lehmann et al. (2021) who reported that 72% of granule-shaped microplastics were aggregate-associated, while 28% were dispersed in agricultural soil. This may be because granule-shaped microplastics cannot be directly produced from plastic film mulching. We found that granule-shaped microplastics measuring 0.25–1 mm were more abundant over time in FM soil aggregates measuring 0.25–2 mm than in comparable NM aggregates, indicating that the size distribution of granule-shaped microplastics may have been related to their morphology in different soil aggregates (Kooi and Koelmans, 2019). This supports the idea that different microplastic particle sizes selectively bind to suitable soil aggregates through microbial biodegradation (De Tender et al., 2017; Xie et al., 2021; Liu et al., 2022) during organic matter and microplastic decomposition (Ganesh Kumar et al., 2020; Xiao et al., 2022; Zhang et al., 2022a, 2022b).

After some durations, microplastics of different shapes and sizes were more abundant in NM aggregates than in FM aggregates or showed similar abundance in both groups. This indicated that mulching was not the only factor affecting microplastic distribution in soil aggregates, and that environmental and human factors should also be considered (Zhang et al., 2022a, 2022b). This could be explained as follows. First, microplastic migration strongly depends on the hydrophobicity of the initial concentration and the characteristics of the surrounding environment (i.e., water, oil, and solvents) (Li et al., 2016). Microplastic contamination can occur owing to organic fertilizer application, sewage irrigation, precipitation, and/or atmospheric deposition (Dris et al., 2016; Nizzetto et al., 2016; Kong et al., 2012). Furthermore, cracked and weathered of film residues present in FM soil may be blown to NM soil by the wind (Li et al., 2022) or flow to other plots through surface runoff (Wei et al., 2021), increasing microplastics in NM soil. This is in line with the findings of Allen et al. (2019) who reported on the relative daily counts of 73 film- and 44 fiber-shaped microplastics collected at a remote mountain site via atmospheric deposition from higher altitudes. Zhang et al. (2022a) also reported that agricultural film waste and residue from vehicle tires (our study site was close to the road) can enter the soil via atmospheric deposition or wind transport pathways. Importantly, local farmers are highly aware of the recyclability of plastic film mulch in FM fields, resulting in low residual film content in FM plots. The above factors could cause fiber-, granule- and film-shaped microplastics to be more abundant in NM soil near FM plots than in the FM plots. This could also explain why microplastic distribution in FM and NM soils varied between years.

This study is only preliminary and investigated the distribution of microplastics of different shapes and sizes in soil aggregates after plastic film mulching. However, it did not consider the effects of combining microbial communities and plants to express the partitioning of microplastics in soil aggregates. Therefore, a more in-depth mechanistic study that also considers the organic matter turnover of microorganisms under different fertilization methods and cultivated crops is required in the future.

5 Conclusion

In this study, long-term soil subjected to FM treatment was associated with decreased SOC content in 0.053–0.25 mm soil aggregates when compared with that in NM soil. The distribution of microplastics was inhomogeneous in soil aggregates between FM and NM plots. Film- and fiber-shaped microplastics in soil aggregates were generally more abundant in FM than in NM soil. In particular, 0.25–1 mm fiber-shaped microplastics in 0.053–0.25 mm soil aggregates, 0.25–1 mm granule-shaped microplastics in 0.25–2 mm soil aggregates, and 0.05–0.25 mm and 1 + mm film-shaped microplastics in 0.25–2 mm soil aggregates were more abundant in FM soil than in NM soil. After some durations of continuous mulching, these trends changed, likely due to natural and human activities. Microplastic accumulation is a concern for the sustainable development of agricultural soil. Focusing on the distribution of microplastics in soil aggregates improves our understanding of the risk that microplastics pose to the environment and help us to maintain soil health and quality.

Abbreviations

FM, Film mulching; NM, No mulching.

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Electronic supplementary material

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