# Polyester Microplastic Fibers affect Soil Physical Properties and Erosion as a Function of Soil Type

Rosolino Ingraffia<sup>1,2,3\*</sup>, Gaetano Amato<sup>1,4</sup>, Vincenzo Bagarello<sup>1</sup>, Francesco G. Carollo<sup>1</sup>, Dario Giambalvo<sup>1,4</sup>, Massimo Iovino<sup>1</sup>, Anika Lehmann<sup>2,3</sup>, Matthias C. Rillig<sup>2,3</sup>, Alfonso S. Frenda<sup>1</sup>

<sup>1</sup>Department of Agricultural, Food and Forest Sciences, Università degli Studi di Palermo, Palermo, Italy;
<sup>2</sup>Plant Ecology, Institute of Biology, Freie Universität Berlin, Berlin, Germany;
<sup>3</sup>Berlin-Brandenburg Institute of Advanced Biodiversity Research, Berlin, Germany;
<sup>4</sup>Fondazione A. e S. Lima Mancuso, Piazza Marina 61, 90133, Palermo, Italy

\*Correspondence to: Rosolino Ingraffia (rosolino.ingraffia@unipa.it)

**Abstract.** Microplastic is recognized as a factor of global change affecting many environmental compartments. Agricultural soils are likely hotspots of microplastic contamination in terrestrial ecosystemsvery likely to receive microplastic contamination and are of particular concern due to their role in food production. Microplastic has already been shown to be able to affect soil properties, but its effect on different soil types is poorly understood. Moreover, nolimited information is available on how the presence of this pollutant can affect soil water erosion processes, which are extremely important issues in many environments. In the light of this, we performed two experiments (a microcosm and a mesocosm studycarried out on a microscale) to investigate how the presence of polyester microplastic fibers affects soil physical and hydrological parameters and processes such as aggregate formation and soil erosion in three different agricultural soil types (a Vertisol, an Entisol, and an Alfisol).

Our data show that the effects of polyester microplastic <u>fibers</u> on soil physical parameters (including soil aggregation and erosion) are strongly dependent on soil type. We found that microplastic <u>fiber</u> contamination can decrease the formation of new aggregates but at the same time increase their stability in water, with effects on soil erosion stronger as the intrinsic erodibility of soil increases affect soil bulk density, capacitive indicators of soil physical quality, and decrease the formation of new aggregates (labile in the incubation period applied in our experiments) but did not affect their stability in water. However, we found that polyester MP fibers reduced soil loss and sediment concentration, especially in the most erodible soils. In this paper we provide some hypotheses but certainly future data are still needed to confirm or disprove them.

Overall, our results highlight the importance of broadly exploring soil properties such as texture, mineralogy, and organic carbon content, etc. to better understand how the various soil types respond to microplastic <u>fiber</u> contamination.

## **1** Introduction

Although the production of plastic is relatively recent ( $\sim$  1950), its versatility and low cost have made it one of the most used daily materials today and it is hard to think of a world without plastic. World production of plastics has exceeded 350 million

tons per year. Only a modest fraction of it is recycled (6-26%; <u>Alimi et al., 2018)</u><u>Alimi et al., 2018</u>) while the vast majority generates plastic waste. <u>Improper disposalMishandling</u> of plastic waste can lead to environmental contamination; and indeed, small plastic particles have been documented even in the most remote areas of the globe (<u>Bergami et al., 2020; Napper et al., 2020)</u>. In the last decade, research on environmental impacts of plastic has received growing attention especially through the study of microplastic (defined as plastic particles smaller than 5 mm; <u>Hartmann et al., 2019</u>).

Most studies on plastic pollution concern aquatic environments, although most plastics are likely present in the terrestrial environment (estimated in quantities 4 to 23 times greater than those in the ocean; Horton et al., 2017). Horton et al., 2017). Given their role in food production, agricultural ecosystems are of particular concern. Microplastics (MPs) can reach agricultural soils through different routes: (i) incorporation of compost, sewage, sludge, <u>coatings of seeds and fertilizers</u>, and other organic compounds often contaminated with MPs ((Weithmann et al., 2018; <u>Zubris Zubris and& Richards</u>, 2005; <u>Richards</u>, 2005; <u>Weithmann Zhang et al. et al.</u> 2018, 2020); (ii) fragmentation of plastic films used for mulching (Qi et al.(Qi et al., 2020) 2020) or (iii) atmospheric fallout (Dris et al., 2018) the soil is generally very slow (O'Kelly et al., 2021), the current hypothesis is that the MP content in agricultural land will slowly increase over time.

Once the MP particles reach the soil surface, they can be easily incorporated into the soil profile through terrestrial fauna-(earthworms: Rillig et al. Rillig et al. 2017a, 2017; Collembola: Maaß et al. Maaß et al., 2017 2017), biopores, or mechanical operations; (e.g., ploughing), or soil cracking (Rillig(Rillig, Ingraffia, et al. et al. 2017b). Therefore, MPs that reach the soil surface can be easily incorporated, 2017), cryoturbation, etc. Incorporated MPs into soil and thuscan interact with soil biophysical processes. Indeed, several studies have shown that the presence of MP can affect soil physical characteristics (bulk density, porosity, aggregate stability, water retention capacity, etc.) with effects widely differentiated in relation to the soil characteristics and MP type, shape and size ((de Souza Machado et al., 2018; Zhang and LiuBoots et al., 2019; Lozano et al., 2021; Machado et al., 2018; \_2018; BootsZhang et al., 2019 et al., 2019). Among the various microplastic types, polyester MP fibers are one of the most detected in agroecosystems (Büks & Kaupenjohann, 2020; ; ZhangCrossman-et al., et al., 2019; Lozano et al., 2021). As these soil characteristics are more or less strictly related to the susceptibility of soil2020) and have been shown to be able to modify markedly (often stronger than other MP types) several soil physical properties. For instance, Machado et al. (2018) found that polyester MP fibers decreased soil bulk density and increased soil water holding capacity in an Albic Luvisol, while Lehmann et al. (2019), in the same soil type, found that this contaminant tends to decrease soil aggregate water-stability.

As soil physical characteristics are related to its susceptibility to erosion (Bradford et al., 1987; Lowery et al., 1995; Mamedov & Levy, 2019)(Bradford et al., 1987; Lowery et al., 1995; Mamedov & Levy, 2019), it is reasonable to expect that microplastics<u>MPs</u> in the soil can influence water erosion processes, which are extremely important issues in many environments (Bagarello et al., 2018)(Bagarello et al., 2018).

\_However, to the best of our knowledge, nolimited research has been conducted to evaluate if and to what extent MP contamination of soil affects water erosion processes.

In light of the above, plastic likely represents a risk for the functionality and resilience of ecosystems. Moreover, due to erosion processes, microplastic particles embedded in the upper soil layer can be transported, eventually reaching water bodies such as rivers, lakes or the sea (Rehm et al., 2021)(Rehm et al., 2021). To better manage this situation, it is necessary to understand the sources, movements, fate, and impacts of plastic when it reaches the different environmental compartments.

The main objective of this research is to expand the knowledge on the effects of MP contamination on the physical processesproperties of three different soil types and evaluate the impact of MPspolyester MP fibers on the extent of water erosion processes. The underlying hypotheses are: <sup>1</sup>/<sub>i</sub>) polyester MP fiber contamination negatively affects the structural state of the soil and increases soil erosion; ii) the adverse impact of microplastic polyester MP fiber contamination on physical soil properties and hydrological processes differs with soil type, with more marked effects in the more "fragile" ones; 2) microplastic contamination, negatively affecting the structural state of the soil, increases the extent of soil erosion soils that are characterised by a low aggregation ability. In these soils the presence of MP fibers could substantially increase the susceptibility to the erosion processes. Collecting this information is essential for identifying the damages and developing solutions that can reduce the impact of microplastics on ecosystems and, at the same time, concentrate efforts in terms of resources and interventions in the most vulnerable areas.

#### 2 Material and Methods

## 2.1 Soils and treatment with microplastics

Three widely different soils were sampled at the end of October 2019 from the upper 30 cm of agricultural fields, classified according to the soil taxonomy and located as follows:

### i) Typic Haploxerert (Vertisol; 37.556140°N, 13.515400°E, 360 m amslpolyester microplastic fiber treatment

For our experiment we chose the following three different soils, widely spread in the Mediterranean area:

i) Typic Haploxerert (Vertisol). This soil type is widespread in Mediterranean environmentsareas with a flat or slightly sloping morphology. The sampled soil has a elay texture (Table 1) is well-structured, with clay texture, a good water and stable structure, anutrient accessibility, sub-alkaline reaction, a fair or high presence of organic matter and other elements of fertility-Large (P, K, N, etc). Smectite (montmorillonite) is the dominant clay mineral. This soil is characterized by large and deep cracks along the profile characterize this soil-during the dry season. It has a medium-high production potential.

ii) Typic Xerorthent (Entisol; 37.561368°N, 13.512904°E, 414 m amsl). It is a light brown type of soil with a sub-alkaline reaction, widespread in the Mediterranean environmentareas with steep and uneven morphologies, frequently affected by erosion phenomena; it generally has a limited thickness with an abundant skeleton presence. It contains illite as the dominant

<u>clay mineral followed by kaolinite.</u> The structure is not very stable, the organic matter content is modest, and the production potential is medium-low.

iii) Typic Rhodoxeralfs (Alfisol; 37.643511°N, 12.628327°E, 38 m amsl). It is a typical soil widespread on the carbonate platforms of many Mediterranean environments; it can also be found in mountain contexts that are carbonated and when located in high slopes they are susceptible to severe erosion. The colour tends to be red due to the considerable presence of iron oxides linked to the leached clays. Kaolinite is the dominant clay mineral. This soil is characterized by strong pedological aridity, due to its calcareous nature, and low amounts of organic matter and elements of fertility content.

fertility elements.

The soils were sampled at the end of October 2019 from the upper 30 cm of agricultural fields. After sampling, the soil was air dried, sieved at 600 µm and stored at 4 °C until the beginning of the experiments in December 2019. This was to minimize changes in the natural microbial community. Each soil was characterized by measuring particle size distribution (Gee et al.Particle size distribution was determined using conventional methods and soil texture was classified according to USDA (Gee & Bauder, 1986), 1986), total. Total nitrogen (TN; Kjeldhal), total organic carbon (TOC; Walkley–Black procedure according to Nelson-Nelson and&-Sommers, 1996) 1996), pH, saturated electrical conductivity at 25 °C (EC), and cation exchange capacity (CEC) (of the considered soils are listed in Table 1).

	Soil	<del>Clay</del>	Silt	Sand	TN	TOC	<del>рН</del>	EC	CEC	
		<del>(g kg <sup>-1</sup>)</del>	<del>(g kg<sup>-1</sup>)</del>	<del>(g-kg<sup>-1</sup>)</del>	<del>(g-kg<sup>-1</sup>)</del>	<del>(g-kg<sup>-1</sup>)</del>		<del>(dS-m<sup>-1</sup>)</del>	<del>(emol kg</del> -	ŀ <b>)</b>
	Vertisol	4 <del>15</del>	<del>357</del>	<del>228</del>	<del>1.54</del>	<del>15.78</del>	7.74	<del>1.89</del>	<del>30.0</del>	
	Entisol	<del>209</del>	<del>461</del>	<del>330</del>	<del>1.20</del>	<del>9.25</del>	7.84	<del>1.88</del>	18.4	
	<del>Alfisol</del>	<del>152</del>	431	417	<del>0.77</del>	<del>11.20</del>	<del>7.58</del>	<del>2.01</del>	<del>13.8</del>	
Soil	Site c	oordinates	Clay	<u>Silt</u>	Sand	TN	TOC	<u>pH</u>	EC	CEC
5011			<u>(g kg<sup>-1</sup>)</u>	<u>(g kg<sup>-1</sup>)</u>	<u>(g kg<sup>-1</sup>)</u>	<u>(g kg<sup>-1</sup>)</u>	<u>(g kg<sup>-1</sup></u>		<u>(dS m<sup>-1</sup>)</u>	(cmol kg <sup>-1</sup> )
Vertisol		<u>56140°N,</u>	<u>415</u>	<u>357</u>	<u>228</u>	<u>1.54</u>	<u>15.78</u>	<u>7.74</u>	<u>1.89</u>	<u>30.0</u>
		<u>13.515400°E,</u>								
Entisol		<u>61368°N,</u>	<u>209</u>	<u>461</u>	<u>330</u>	<u>1.20</u>	<u>9.25</u>	<u>7.84</u>	<u>1.88</u>	<u>18.4</u>
	<u>13.5</u>	<u>12904°E</u>								
<u>Alfisol</u>	<u>37.6</u>	<u>43511°N,</u>	<u>152</u>	<u>431</u>	<u>417</u>	<u>0.77</u>	<u>11.20</u>	<u>7.58</u>	<u>2.01</u>	<u>13.8</u>
<u></u>	<u>12.6</u>	<u>28327°E</u>								

Table 1. Physical and chemical properties of the three different soils used in the experiment.

<u>Clay, Silt, and Sand were classified according to USDA (Clay < 2μm, Silt 2-50 μm, and Sand 50-2000 μm);</u> TN, Total Nitrogen; TOC, Total Organic Carbon; EC, Electrical Conductivity; CEC, Cation Exchange Capacity.

For microplastic contamination, a 100% polyester white rope (Marlow, Marlowbraid classic rope) was manually cut to produce secondary microplastics microplastic fibers. These fibers were characterized by scanning at least 200 fibers on PVC travs 10 times (Epson Perfection Scan V800, 8-bit grayscale, 800 dpi) and then analyzing the scans with WinRhizo (WinRHIZO Pro v. 2007d, Regent Instrument Inc., Quebec, Canada). The mean and the standard deviation, SD, of fiber length were 2.87 mm and 0.31 mm, respectively, and the mean and SD of fiber diameter were 87 um and 3 um, respectively. We chose to use polyester fiber because 1) fibers represent a main form of microplastic pollution in soils (Liu et al, 2018); 2) among these, polyester is one of the most represented materials (Crossman et al, 2020) and seems to cause marked effects on soil biophysical characteristics (de Souza Machado et al, 2018). The polyester fibers were incorporated into the soil at a concentration of 0.5% w/w of the dry soil weight. This microplastic level was similar to that used in previous studies, which reported noticeable changes in the soil biophysical environment and plant response <del>((de Souza Machado</del>Lehmann et al., 2020; Lozano-et al et al., , <del>2018 and 2019; Lozano</del>2021; Machado et al., 2018; Machado-et al et al., <del>2020; Lehmann</del>2019; Ingraffia-et al et al., <del>2020).</del> The2022). To contaminate the soils, the fibers were added into the blender (Waring® WSG30, Waring Commercial, Torrington, Connecticut) as a band sandwiched between two layers of soil. We chose to incorporate the fibers into the soil using a blender to provide a more homogeneous distribution of the fibers in the soil. We tested the impact of mixing time to establish a protocol which ensured a homogeneous distribution of the polyester MP fibers into the soil, and that preserved the integrity of the MP fibers (which was evaluated through visual inspection using a stereo microscope Zeiss Stemi 2000-C; Fig. 1a and b). The mixture of soil and fibers was mixed five times for  $\frac{555}{58}$  s each. The same disturbance was also applied to the soil of the control treatment.

The three soil types, both treated and untreated, were used to fill 36 cylinders in total (height = 0.05 m, diameter = 0.05 m; 80 g of soil; Fig. <u>lalc</u>) to evaluate the impact of microplastic on soil properties (experiment I) and 36 <u>runoff plotssoil trays</u> of  $0.30 \times 0.15 \times 0.10$  mm<sup>3</sup> perforated at the bottom (Fig. <u>lbld</u>) for the rainfall simulation tests (experiment II).

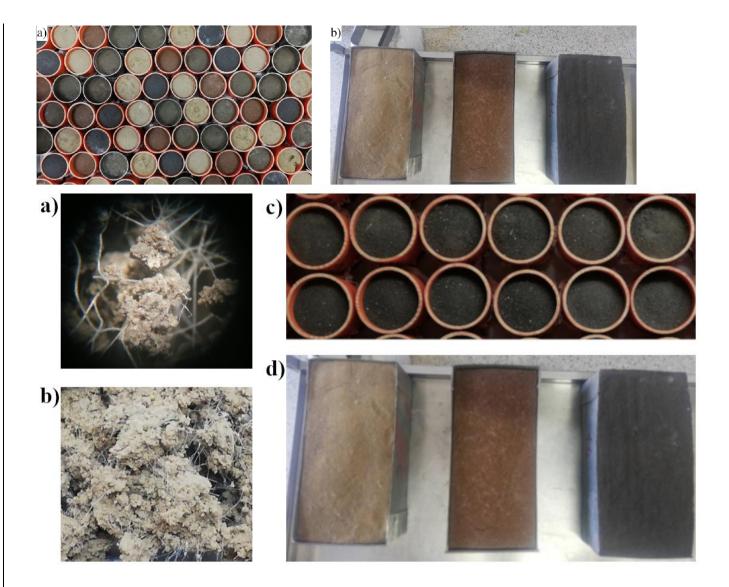


Figure 1. a) Cylinders and b) runoff plots used in the experimentscontaminated soils; c) experimental units Experiment I; d) experimental units Experiment II.

For both treatments, that is the control (non-treated, Ctr) and the microplastics treated (MP) soils contaminated with polyester MP fibers, the soil samples were watered with distilled water to near field capacity by capillarity and then incubated in a growth chamber in the dark at  $23 \pm 2$  °C and  $60 \pm 5$  % relative humidity for nearly 6 months. During the incubation period, the soil samples were watered once a week with distilled water to field capacity by capillarity. The experimental units were re-randomized at each irrigation event. It was not possible to follow this protocol for 35 days (from April 7 to May 15, 2020) because of the national lockdown due to the COVID-19 pandemic. However, the status of the experimental units was checked after this period and no visual damage was observed.

#### 2.2 Experiment I. Impact of polyester microplastic fibers on soil properties

For each soil type, the Ctr treatment was set up in 8 replicates while the MP treatment consisted of 4 replicates (for a total of 36 cylindrical samples).

The soil water retention curve was determined after incubation using the hanging water column apparatus (Dane <u>(Dane and Hopmanset al., 2002a)</u>, 2002a) for pressure head, h, values ranging from -0.03 to -1 m and the pressure plate extractor (Dane <u>(Dane and Hopmanset al., 2002b)</u>, 2002b) for h values ranging from -3.3 to -150 m.

Briefly, the soil samples were placed on the porous plate of a glass funnel and saturated from the bottom by progressively raising the water level in a graduated burette (height was adjustable). Equilibration at *h* values of -0.2, -0.1, and -0.05 m was obtained successively in steps of 24 hours each. Finally, the samples were fully saturated by submersion for 2 hours. From saturation, soil samples were desorbed by imposing a sequence of eight *h* values (h = -0.03, -0.05, -0.10, -0.20, -0.30, -0.50, -0.70, and -1.0 m). At each *h* level, the volume of drained water into the burette was recorded. The volumetric water content,  $\theta$  (m<sup>3</sup>m<sup>-3</sup>), at each equilibrium stage was calculated by adding the drained volumes to the value determined atfinal  $\theta$  value corresponding to h = -1 m after oven-dryingthat was determined by oven-drying the samples at 105°C. The dry soil bulk density, *BD* (g cm<sup>-3</sup>), was calculated by measuring the the measured volume at the end of the experiment (i.e., h = -1 m) and using the oven-dried weight of the soil sample. Given that polyester can withstand temperatures as high as 150°C and the added amount of fibers (0.5%) was low, the drying process was not expected to have a significant effect on the measured *BD* values.

Then, the oven-dried soil samples were gently crushed and split into two sub-samples: one was used to determine the soil water content down to -150 m, while the other was used to determine the soil structure and the soil water-stable aggregates.

To determine the soil water content at lower pressure head values, the soil was packed to the same *BD* value of the larger samples in rings with a diameter of 0.05 m and a height of 0.01 m. These repacked soil samples were then used to determine the soil water content corresponding to h = -3.3, -10, -33 and -150 m using the pressure plate apparatus (Dane-(Dane and Hopmanset al., 2002b), 2002b).

The water retention model proposed by van Genuchten (1980)van Genuchten (1980) was then fitted to experimental data obtained for each soil sample and the. The water content values corresponding to saturation,  $\theta_s$ , and to the pressure heads h = -0.10, -1.0, and -150 m ( $\theta_{-0.1}$ ,  $\theta_{-1.0}$ ,  $\theta_{-150}$ ) were then estimated from the model. The following "capacitive" indicators of soil physical quality were considered that are directly linked to the soil water retention properties (<u>(ReynoldsIovino et al., et al., 2009; Iovino2016; Reynolds et al., et al., 2016)2009</u>):

Macroporosity 
$$P_{mac} = \theta_s - \theta_{-0.1}$$
 (1)  
Air capacity  $AC = \theta_s - \theta_{-1.0}$  (2)

$$PAWC = \theta_{-1.0} - \theta_{-150} \tag{3}$$

The  $P_{mac}$  indicator gives the volume of large (macro) pores (i.e. > 0.3 mm equivalent pore diameter), which indicates the soil's ability to quickly drain excess water and facilitate root proliferation (Reynolds et al., 2009)(Reynolds et al., 2009). The AC index represents the ability of soil to store and provide essential soil air (Topp et al., 1997)(Topp et al., 1997). The PAWC indicator is a measure of the ability of the soil to store and provide soil water that is available to crop roots.

To investigate soil structureaggregation and soil water-stable aggregates, we used a wet sieving apparatus (Retsch AS 200) with a set of stacked sieves mesh of 2 mm, 1 mm, 0.6 mm, 0.5 mm, 0.25 mm, and 0.106 mm. For the soil structureaggregation, we closed the water valve and we performed a dry sieving using a 40 g subsample of air-dried soil from each treatment; the sieves were moved vertically at rate of 30 cycles per minute over a period of 3 minutes, and after recording the weight of each sieved fraction, the whole sample was reconstructed and used to assess water-stable aggregates by using the same procedure with the opened water valve. As the soil at the beginning of the experiment was sieved at 600  $\mu$ m, this method allowed us to also determine how our treatments affected the formation of new aggregates (> 600  $\mu$ m) and the stability of these in water. All the analyses were conducted in a laboratory under controlled conditions of temperature (mean T = 22°C).

Data obtained from dry and wet sieving were used to calculate:

1) percentage of newly formed aggregates (> 600 µm; NFA) according to the following equationLehmann et al. (2021):

$$\frac{NFA(\%)}{P_{sample}} = \frac{A_{dry>600}}{P_{sample}} \times \frac{100}{NFA(\%)} = \frac{W_{>600}}{W_{sample}} \times 100$$
(4)

where  $A_{dry}W_{>600}$  is the weight of the >600 µm fraction after air dried or wet sieving process,  $P_{sample}W_{sample}$  is the total weight of the sample.

percentage of water-stable newly formed aggregates (>600  $\mu$ m; WSNFA) – calculated as following: As the soil at the beginning of the experiment was sieved at 600  $\mu$ m, this metric represents the formation of new macro-aggregates (> 600  $\mu$ m) certainly built from smaller size aggregates representing a shift in aggregate size distribution.

 Mean weight diameter (MWD; mm) calculated on data derived from dry (MWD<sub>dry</sub>) and wet (MWD<sub>wet</sub>) sieving using the following equation (Deviren Saygin et al., 2012):

$$\frac{WSNFA(\%)}{W_{dry>600}} = \frac{A_{wet>600}}{A_{dry>600}} \times \frac{100}{MWD} = \sum_{i=1}^{7} \frac{W_i}{W_{sample}} \times d_i$$
(5)

where  $A_{wee}>600$  is the weight of the >600 µm fraction after the wet sieving process. 3) water stability index (*Simdex*) – calculated as following:

$$S_{index} = 100 - \frac{MWD_{wet}}{MWD_{dry}} \times 100 \tag{6}$$

where *MWD*<sub>wer</sub> and *MWD*<sub>dry</sub>, are the mean weight diameter after wet and air dried sieving processes respectively, calculated as follows,

$$\underline{MWD} = \sum_{i=1}^{7} \quad \frac{A_i}{P_{sample}} \times d_i \tag{75}$$

where *i* is the fraction size (> 2; 1-2; 1-0.6; 0.6-0.5; 0.5-0.25; 0.25-0.106; < 0.106 mm),  $A_i W_i$  is the oven-dry weight of each fraction after sieving process (dry and wet);  $d_i$  is the mean diameter of each size fraction (i.e. the mean inter-sieve size; for the fraction > 2.0 mm<sub>a</sub> (that was extremely lowsmall and always less than 1%)<sub>a</sub> it was considered 2.2 mm).

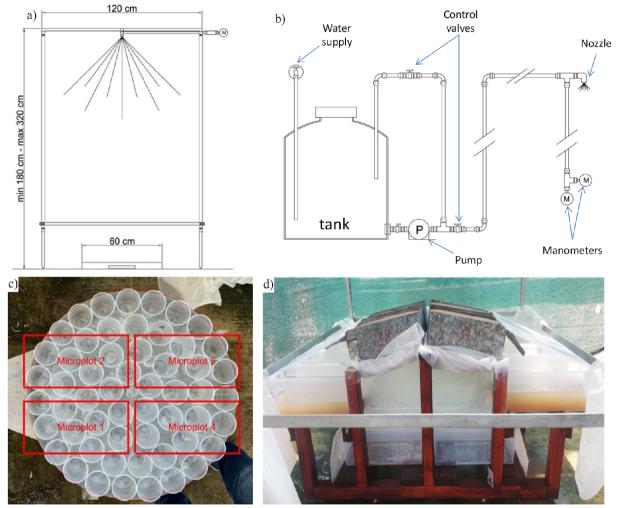
#### 2.3 Experiment II. Rainfall simulation

Soil with (MP) and without (Ctr) microplasticspolyester MP fibers was used to perform the experiments with the rainfall simulator. For a given treatment, air dried soil was used to fill the runoff plotsoil tray after placing a cotton guard cloth at its bottom to avoid soil loss through the draining holes. The soil was gradually poured into the runoff plottray and it was compacted manually by repeatedly dropping the runoff plottray from a height of approximately 0.05-0.10 m until soil compaction ceased. Therefore, eachEach soil sample had an initial bulk volume of  $0.30 \times 0.15 \times 0.10 = 4.50 \times 10^{-3}$  m<sup>3</sup>. For a given soil and a given treatment (MP and Ctr), six runoff plotssoil trays were prepared (total sample size, N = 3 soils × 2 treatments × 6 runoff plotssoil trays = 36).

The rainfall simulator is a nozzle type rainfall simulator, very similar to those described by RiesIserloh et al. (et al. (20092012) and ) and IserlohRies et al. (et al. (20122009)). The major parts of the rainfall simulator (Fig. 2a) are: a square metal frame (120 cm  $\times$  120 cm) with a nozzle located at its centre and four telescopic steel legs in order to position the nozzle at a height ranging from 180 to 320 emdifferent heights above the ground surface. A tank ensures the supply of water which is pressurized by a low pressure 12 V bilge pump (model Shurflo 2088 713-515) (Fig. 2b). The water supply to the tank assures a steady flow. The flow rate is regulated by the control valve located on a recirculation circuit and it is checked by a digital manometer (model PCE- DMM 10) characterized by an accuracy equal to  $\pm$  0.5%. In order to check the pressure measure, a further analog manometer is installed.

Rainfall intensity and its uniformity over the wetted area were checked before performing the runoff and soil loss measurement experiment. Initially, a total of 55 plastic cups, each with an upper surface area of 35.3 cm<sup>2</sup>, were placed on a circular metal frame, with a diameter of 60 cm, that was placed at a distance from the nozzle equal to the average distance between the nozzle and the runoff plots and centeredsoil trays and centred exactly below the nozzle of the rainfall simulator (Fig. 2c). Simulated rainfall was collected for 30 min and a rainfall intensity value was then determined for each cup. Taking into account that four plotstrays were planned to simultaneously be subjected to a given rainfall event, rainfall uniformity among these plotstrays was verified. In particular, the data obtained on the cups corresponding to each plottray were averaged to obtain four mean rainfall intensity values, that is a value to be associated with each plotsoil tray. Rainfall uniformity was assessed by calculating the following uniformity coefficient, CU (Christiansen, 1942)(Christiansen, 1942):

$$CU = 100 \left( 1 - \frac{\sum_{i}^{n} |R_i - R|}{nR} \right) \tag{86}$$



being n = 55 the number of cups,  $R_i$  = the rainfall intensity recorded by each cup and R = the mean rainfall intensity.

**Figure 2.** a) Scheme of the rainfall simulator and b) the hydraulic setup; c) arrangement of the plastic cups to evaluate rainfall intensity uniformity and overlap between the plastic cups and the <u>micro-plotssoil trays</u> (in red); d) view of the experimental <u>runoff plotsoil trays</u> during a rainfall simulation event.

Each individual rainfall simulation experiment was carried out by placing four <u>runoff plotssoil trays</u> on a frame imposing a steepness of 15% to each <del>plot</del>try (Fig. 2d). This steepness value was chosen to consider a potentially favourable condition to appreciable soil erosion processes and also in the perspective to replicate the experiment with natural rainfall events and larger plots in the future. This new experiment could be performed at the Sicilian Sparacia experimental area, where micro-plots and plots were established on a 14.9% slope (Bagarello & Ferro, 2004). For each <del>plots</del> runoff and the associated sediment load was conveyed through a gutter to a plastic container and the same was done with reference to deep percolation.

Lids and plastic sheets were used in order to only expose soil surface to rainfall during the experiment. The <u>runoff plotsoil tray</u> with the soil was weighed before and immediately after the rainfall event to obtain the change in the stored water in the soil volume. The <u>nozzle was placed at a mean height of 1.70 m (measured from the middle of each tray). The</u> whole experiment was carried out with a mean rainfall intensity of 33.4 mm h<sup>-1</sup> (CV = 3.0%) and it had a duration offor 2 hours. The rainfall simulation experiment was completed in nine days. At the beginning of each working day, rainfall intensity was measured by collecting water for ten minutes in a cylindrical bucket with a diameter of 30 cm, and at a distance from the nozzle equal to the average distance between the <u>runoff plotsnozzle</u> and the <u>nozzlesoil trays</u>.

At the end of the experiment, all containers were transported to the laboratory and dried at 105 °C to constant weight to determine the total surface runoff volume, the total drained water volume and the associated load of solids. For the drained water volume, this last quantity was always small and practically under the detectable quantity. Therefore, drained water was essentially clear in all cases. The data were then used to calculate, for each <u>plotsoil tray</u>, total runoff per unit area,  $V_e$  (mm), total percolation per unit area,  $P_e$  (mm), total soil loss per unit area,  $A_e$  (g/m<sup>2</sup>), and sediment concentration in the surface runoff volume,  $C_e$  (g/m<sup>3</sup>). A check of the reliability of the  $V_e$  and  $P_e$  data was made, taking into account that the sum of total runoff, total percolation and change in the stored water in the soil volume, denoted as  $S_e$  (mm), was expected to nearly coincide with the rainfall depth for the simulated event,  $h_e$ .

#### 2.4 Statistical analysis

The data of both experiments were analyzed in R (R Core Team, 2020)(R Core Team, 2020) using a generalized least square models in the "nlme" package (Pinheiro et al., (Pinheiro et al., 20182021)) with implemented varIdDent() function to account for heterogeneity of variance. Model residuals were checked for heteroscedasticity and normal distribution.

All response variables within each soil type were compared between the two groups (MP minus Ctr) using the package "dabestr" (Ho et al., 2019), 2019) to calculate the effect sizes as unpaired mean differences and generate a biascorrected and accelerated bootstrapped 95% Confidence Intervals (CIs). We used this combined approach based on the expanding recognition of the limitation of using only "P-value statistic" approach and avoid dichotomous cutoffs (Ho et al.(Ho et al., 2019; Wasserstein, 2019; Wasserstein and&-Lazar, 2020) Lazar, 2020).

#### **3** Results

#### 3.1 Experiment I

The physical and hydrological effects of the treatment with <u>microplasticspolyester MP fibers</u> differed with the soil type (Table S1). In particular, for the Vertisol, <u>microplasticpolyester MP fibers</u> caused a decrease of bulk density (-9%;), <u>and</u> an increase in air capacity (+ 34%; Fig. 3) whereas no effect was found on soil macroporosity and plant available water content (Fig. 4). In the Entisol, the contamination with <u>MPpolyester MP fibers</u> did not cause any evident <u>effectseffect</u> on the physical and

hydrological characteristics. In the Alfisol, microplastic polyester MP fibers did not exert effects on the BD but induced pronounced decreases in both the AC (- 26%) and the  $P_{mac}$  (- 85%) and an increase in the PAWC (+ 19%).

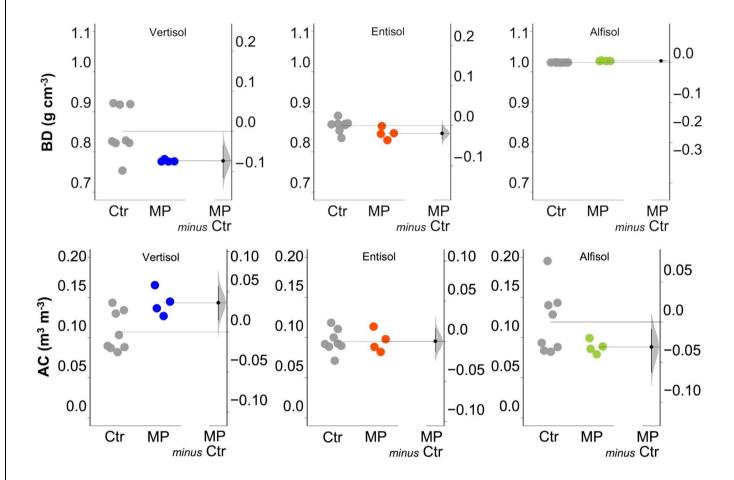
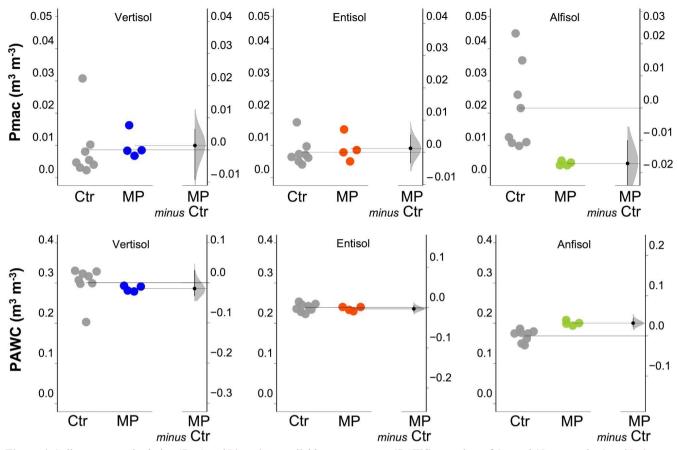


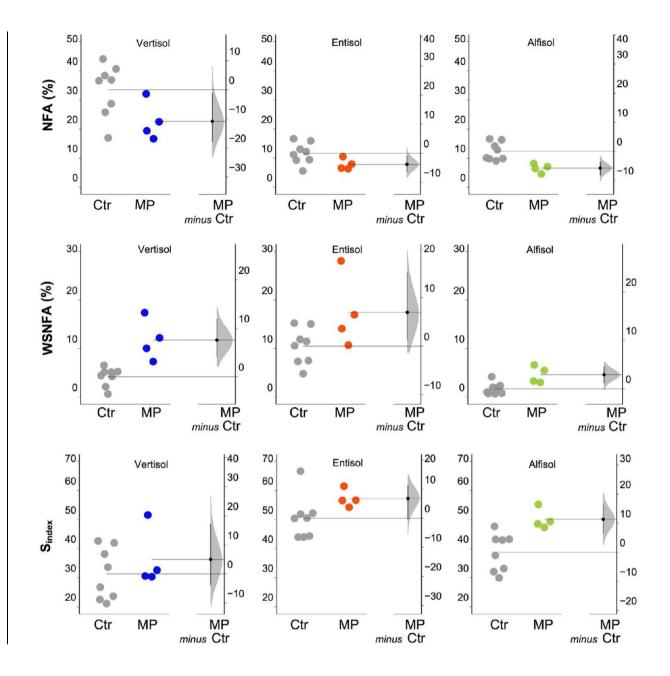
Figure 3. Soil bulk density (BD) and Airair capacity (AC): raw data of Control (Ctr, grey dots) and Polyester microfiberspolyester MP fibers added treatment (MP-add, colored\_colorued dots) are shown in the plot for each soil. The filled curve indicates the resampled distribution of mean differences ( $\Delta$ ), given the observed data. Horizontally aligned with the mean of the test group,  $\Delta$  is indicated by the black circle. The 95% confidence interval of  $\Delta$  is illustrated by the black vertical line.

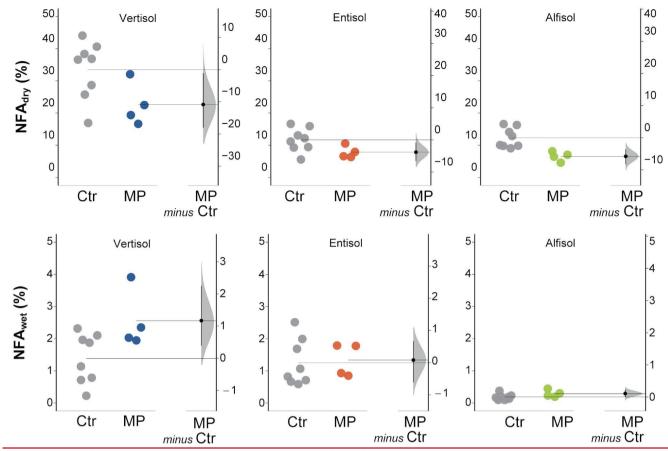


**Figure 4.** Soil macroporosity index ( $P_{mac}$ ) and <u>Plantplant</u> available water content (*PAWC*): raw data of Control (Ctr, grey dots) and <u>Polyester</u> microfiberspolyester <u>MP</u> fibers added treatment (MP-add, <u>coloredcoloured</u> dots) are shown in the plot for each soil. The filled curve indicates the resampled distribution of mean differences ( $\Delta$ ), given the observed data. Horizontally aligned with the mean of the test group,  $\Delta$  is indicated by the black circle. The 95% confidence interval of  $\Delta$  is illustrated by the black vertical line.

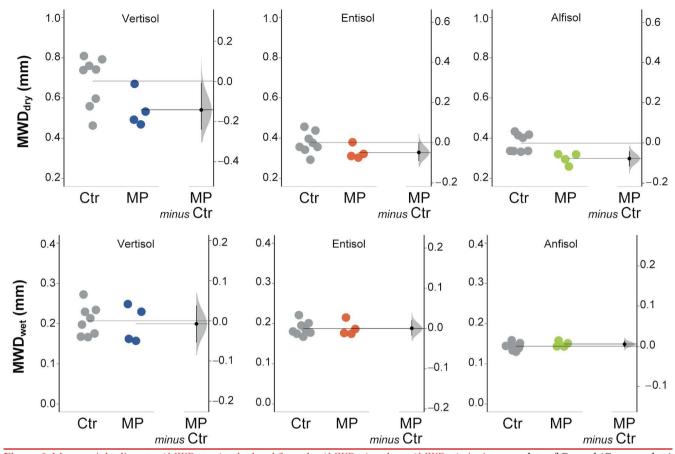
The data obtained from sieving (both dry and wet)dry sieving show how the contamination with microplastics haspolyester <u>MP fibers</u> negatively affected the percentage of newly formed aggregates (> 600  $\mu$ m) (-32% in Vertisol; -3347% in Alfisol; -47% in Entisol;) but has, at the same time, increased their stability in water (+ 180% in Vertisol; + 66% in Alfisol; + 170% in Entisol; Fig. 5).

The water stability index, calculated considering all the classes of aggregates, also increased due to the contamination with microplastics, although with different magnitude depending on soil type (Fig. 5Entisol; -33% in Alfisol; Fig. 5). Only a small fraction of the newly formed aggregates was stable to the subsequent wet sieving (Fig. 5). In addition, MP had a minimal positive effect on NFA<sub>wet</sub> for the Vertisol but it was uninfluential for the Entisol and the Alfisol (Fig. 5). Polyester MP fibers contamination determined a general decrease of MWD<sub>dry</sub> (-21% in Vertisol and Alfisol; -13% in Entisol; Fig. 6), while no effects due to polyester MP fibers were observed on MWD when soil samples were wet sieved (Fig. 6; Tables S2 and S3).





**Figure 5.** Newly formed aggregates (> 0.6 mm; NFA), Water stable newly formed aggregates (>0,6 mm; WSNFA), and Water stability index ( $S_{index}$ )600 µm) calculated from dry ( $NFA_{dry}$ ) and wet ( $NFA_{wet}$ ) sieving: raw data of Control (Ctr, grey dots) and polyester MP fibers added treatment (MP, coloured dots) are shown in the plot for each soil. The filled curve indicates the resampled distribution of mean differences ( $\Delta$ ), given the observed data. Horizontally aligned with the mean of the test group,  $\Delta$  is indicated by the black circle. The 95% confidence interval of  $\Delta$  is illustrated by the black vertical line.



**Figure 6.** Mean weight diameter (*MWD*; mm) calculated from dry (*MWD*<sub>dry</sub>) and wet (*MWD*<sub>wet</sub>) sieving: raw data of Control (Ctr, grey dots) and <u>polyester MP fibers</u> added treatment (MP add, <u>colored</u> dots) are shown in the plot for each soil. The filled curve indicates the resampled distribution of mean differences ( $\Delta$ ), given the observed data. Horizontally aligned with the mean of the test group,  $\Delta$  is indicated by the black circle. The 95% confidence interval of  $\Delta$  is illustrated by the black vertical line.

## 3.2 Experiment II

#### 3.2.1 Preliminary check of the simulated rainfall

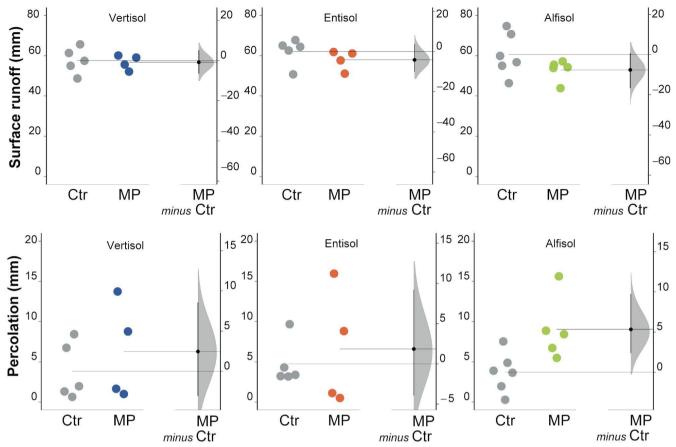
Rainfall intensities, *RI*, measured by the 55 cups ranged from 31.3 to 37.4 mm h<sup>-1</sup>, with a mean of 33.8 mm h<sup>-1</sup>, a coefficient of variation, CV = 4.7% and a uniformity coefficient, *CU*, equal to 96.1%. Considering only the cups corresponding to the area of the runoff plotssoil trays, RI = 33.1 mm h<sup>-1</sup> and CU = 97.1% were obtained. At the individual runoff plotsoil tray scale, the mean rainfall intensity varied from 32.5 to 34.0 mm h<sup>-1</sup>, depending on the runoff plottray, and the *CU* values were in the range 96.8-98.2%. As compared with the performances of other rainfall simulators (Iserloh et al., 2013) 2013), these results suggested that rainfall intensities were reasonably uniform among the four simultaneously tested runoff plotssoil trays and also on each individual runoff plottray.

Mean rainfall intensities were overall stable in the nine days of the rainfall simulator experiment (from 32.1 to 34.9 mm h<sup>-1</sup>). The whole experiment was carried out with a mean rainfall intensity of 33.4 mm h<sup>-1</sup> (CV = 3.0%) and it had a duration of 2 hours. Therefore, the mean rainfall depth,  $h_e$ , was 66.8 mm.

On average,  $h_e - (S_e + V_e + P_e)$  was equal to 5.3 mm. However, neglecting the seven cases characterized by values of  $h_e - (S_e + V_e + P_e)$  greater than 20 mm, for which some anomaly occurred in the experiment, the mean of the remaining 29 values of  $h_e - (S_e + V_e + P_e)$  is very close to zero (0.14 mm). Considering the consistency between  $h_e$  and  $(S_e + V_e + P_e)$ , the seven suspect runs were excluded from the considered dataset to check soil treatment effect on  $V_e$ ,  $P_e$ ,  $A_e$ , and  $A_e$ - $C_e$ 

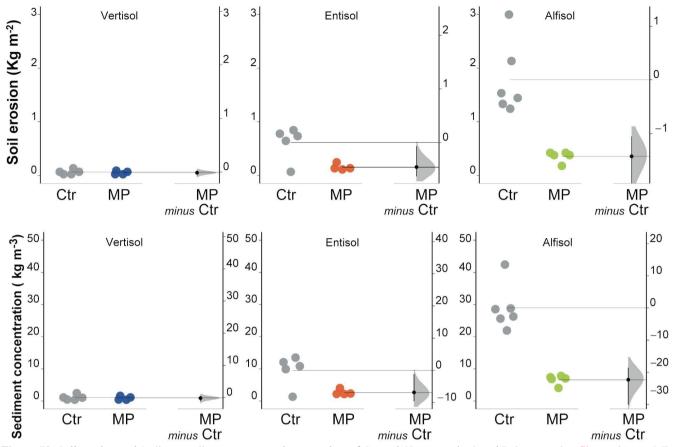
#### 3.2.2 Runoff, percolation, soil erosion and sediment concentration

Soil microplastic polyester MP fibers contamination did not induce changes in surface runoff (Fig. 67) in all soil types, although a slight decrease of  $V_e$  was perceived for both the Entisol and the Alfisol. However, it affected percolation, which varied with the soil type. In particular, the analysis carried out separately by soil type allowed to highlight some different responses of the soils under evaluation; in the Alfisol, the contamination with microplastic polyester MP fibers increased percolation by 144% (Fig. 67) as compared with the Ctr treatment. No effects were observed in the Vertisol and in the Entisol.



**Figure 67.** Surface runoff and <u>Percolationpercolation</u>: raw data of Control (Ctr, grey dots) and <u>Polyester microfiberspolyester MP fibers</u> added treatment (MP-add, <u>coloredcoloured</u> dots) are shown in the plot for each soil. The filled curve indicates the resampled distribution of mean differences ( $\Delta$ ), given the observed data. Horizontally aligned with the mean of the test group,  $\Delta$  is indicated by the black circle. The 95% confidence interval of  $\Delta$  is illustrated by the black vertical line.

The extent of soilSoil erosion varied widely among the soils under evaluation, resulting in a decidedly limited amount in Vertisol and in a particularly high one in Alfisol (Fig. 78). Contamination with microplasticspolyester MP fibers did not cause variations in the quantitiesamount of eroded soil in the Vertisol, while it resulted in substantial reductions in both Entisol (-75%) and Alfisol (-80%). Similar results were observed for sediment concentration, as shown in Figure 78.



**Figure 78**. Soil erosion and <u>Sedimentsediment</u> concentration: raw data of Control (Ctr, grey dots) and <u>Polyester microfiberspolyester MP</u> <u>fibers</u> added treatment (MP-add, <u>coloredcoloured</u> dots) are shown in the plot for each soil. The filled curve indicates the resampled distribution of mean differences ( $\Delta$ ), given the observed data. Horizontally aligned with the mean of the test group,  $\Delta$  is indicated by the black circle. The 95% confidence interval of  $\Delta$  is illustrated by the black vertical line.

## **4** Discussion

As hypothesized, the contamination with microplasticspolyester MP fibers generally resulted in differentiated effects on soil structureaggregation and hydrological characteristics among the tested soils. In the Alfisol polyester microfibers have exerted the most marked effects, enhancing the water holding capacity, reducing the formation of new aggregates and macroporosity and inducing an increase in water stable aggregates. On the contrary, the Entisol was less affected by the presence of microplastics in terms of the physical and hydrological characteristics of the soil. Our results show that MP effects on soil structure and hydrological characteristics are related to the different characteristics of the soil used in the experiments, as already hypothesised by Zhang et al. (2019) and Xu et al. (2020). The results of this study, considering that the Entisol and the Alfisol studied do not differ substantially in particle size composition, highlight how the different response to MP contamination likely do not just depend on the soil texture. Other factors such as the different nature of their particles, and therefore the matrix from which they originated, can alter the interactions that are established between the particles themselves

and polyester microfibers. This deserves further investigation to clarify the mechanisms causing the different responses observed.

In the Vertisol, contamination with polyester microplastic fibers resulted in a decrease in soil bulk density and an increase in air capacity; on the contrary, in the other two studied soils, no variation in soil bulk density was observed. The data available in the literature about MP impact on soil bulk density are limited and not always concordant. For instance, de Souza Machado et al. (2018, 2019) observed a decrease in bulk density in loamy sandy soils of increasing magnitude with increasing concentration of polyester microfibers while Zhang et al. (2019) in a study conducted on a clay loam soil (in field and greenhouse conditions) found no differences in soil bulk density.

Our results show how the presence of MP changes the cohesion between aggregate-forming particles reducing the formation of new aggregatesparticular, the presence of polyester MP fibers determined a decrease of the soil bulk density on the Vertisol, a slight decrease on the Entisol, and no effects on the Alfisol. The data available in the literature about the impact of polyester MP fibers on soil bulk density are limited and not always concordant. For instance, Machado et al. (2018 and 2019) observed a decrease in bulk density of a loamy sand soil at increasing concentration of polyester MP fibers while Zhang et al. (2019) in a study conducted on a clay loam soil (in field and in greenhouse conditions) found no differences in soil bulk density.

Polyester MP fiber contamination negatively affected air capacity and macroporosity index of the Alfisol, maybe as a consequence of the smaller mean diameter of the newly formed aggregates. Given that the BD of this soil was unaffected by polyester MP fiber contamination, it can be supposed that total porosity was redistributed from macropores to meso/micropores as confirmed by the increase of PAWC. No modification of the capacitive indicators of soil physical quality was observed on the Entisol. For the Vertisol, the increase of both total porosity (decrease of *BD*) and *AC* suggested that MP addition favoured macroporosity. Meso/micropores did not change appreciably, as indicated by the similar *PAWC* results for the Ctr and MP treatments.

Our data obtained from dry sieving show that the presence of polyester MP fibers changed the cohesion between aggregateforming particles with a general reduction (although with different effect size on the three soil types) of the formation of new macro-aggregates (> 600 µm). Similar results were observed reported by Boots et al. (2019) and Lozano et al. (2019) and Lozano et al. (2021) (2021). Zhang Zhang and&-Liu (2018) Liu (2018) found that the abundance of aggregate-associated plastic fibers is greater in the micro-aggregates than in the macro-aggregates and this, according to the same authors, would suggest that the presence of fibers in the micro-aggregate limits their possibility to be combined into macro-aggregate. de Sosa Machado et al. (2018) (2018) found that contamination of a loamy-sand soil with polyester MP fibers decreased the fraction of soil forming dry aggregates larger than 1 mm but at the same time increased the formation of large soil clumps and therefore potentially provided additional macro-structures, absent in the non-contaminated soil. In contrast, Zhang et al. (2019)Zhang et al. (2019) found that polyester microplastieMP fibers improve soil aggregation helping to entangle soil particles more efficiently to form aggregates. Interestingly, in this experiment, the effects of MP in soil aggregation varied among soil types highlighting how some soil characteristies (soil mineralogy, elay content, organic matter content, etc.) can influence its effects on the formation of soil aggregates. In particular, the negative effects induced by the addition of MP on the aggregation capacity of the soils were, to a certain extent, of increasing magnitude with increasing TOC;negative effects induced by the addition of polyester MP fibers on the aggregation capacity of the soils were, to a certain extent, of a decreasing magnitude as TOC increased (Vertisol>Alfisol>Entisol; according to the results of the dry sieving; Fig. 5, *NFAdry*; Table 1). Therefore, it would therefore seem that the addition of <u>MPpolyester MP fibers</u> interfered with the formation of macroaggregates by altering the binding mechanism in the soil and, in particular, by reducing the aggregation capacity of organic matter. In particular, high organic matter levels made the MP contamination effects smaller. Similar results were also observedobtained by Liang et al. (2021)Liang et al. (2021) who found that the effects of microplasticpolyester MP fibers on soil aggregation were organic matter dependent. However, in our study the contamination of soils with polyester fibers, in addition to

The presence of the contaminant, although determining a general decrease in newly formed aggregates, induced a general increase in the water-stable aggregate profile; this effect appeared more marked in the clay soil (Vertisol) than in the two loam soils. Also, Zhang et al. (2019), in experiments conducted both in pots and in the field, did not affect the formation of water stable aggregates, since we generally did not observe any appreciable effect of the polyester MP fibers on the MWD and NFA values obtained with the wet sieving. In contrast, Zhang et al. (2019) observed a significant increase in the water of stability of the macro-aggregates (> 2 mm) in water and in the volume of the macro-pores (> 30 µm) after addition of polyester microfibers/MP fibers in a clay soil. In contrast, de SosaOn the other hand, Machado et al. (2018) Machado et al. (2018) found a significant decrease in water-stable aggregates with increasing polyester MP fiber concentration in a sandy- loam soil. Evidently, soil mineralogy can be an important determinant for understanding properties play an important role in guiding the effects of MPspolyester MP fibers in the formation of macro-aggregates and their water stability. This suggests that the different soil particles may have a different affinity for the polyester fibers used here. The interactions that can occur between MP and soil fine particles are still poorly studied; acquiring this information is undoubtedly important in identifying which factors and mechanisms can influence the The interactions that can occur between polyester MP fibers and fine soil particles are still poorly studied. Our results show that the effects of polyester MP fibers on soil structure and hydrological characteristics are most probably related to the different characteristics of the soil used in the experiments, as already hypothesized by Xu et al. (2020) and Zhang et al. (2019). Our three soil types differed for various characteristics (e.g. texture, clay mineralogy, TN, TOC, CEC) which could have played a role in the observed responses to the contamination. Certainly, it is very difficult to identify how and on which factor/s polyester MP fibers influenced our results, also considering the several biotic and abiotic factors that interplay in the process of soil aggregation. For instance, our three soil types widely differ for clay mineralogy: montmorillonite in the Vertisols, illite in the Entisols, and kaolinite in the Alfisols. These three clay types largely differ for size, shape, specific surface area, and structure, and stability of soils contaminated with MP.

Microplastic contamination did not affect surface runoff and drainage in the Vertisol and in the Entisol. It did cause a reduction of both parameters, as a consequence, for their ability to form aggregates (Lal & Shukla, 2004). Therefore, it is possible to presume that polyester MP fibers could have differentially affected the ability of the above-mentioned clay types to form aggregates during the incubation period. Those aspects deserve further investigation to clarify the mechanisms causing the different responses observed.

Polyester MP fiber contamination did not affect surface runoff and drainage in the Vertisol and in the Entisol although a slight decrease of  $V_e$  was perceived for this last soil. However, it caused a slight decrease of surface runoff and an increase of percolation in the Alfisol, which has the coarsest texture among the three tested soils (Table 1). Moreover, the three studied soils differed widely in their susceptibility to erosion (Vertisol < Entisol < Alfisol): this. This result was partly expected, as many authors have highlighted how the increase in silt content and the reduction in organic matter contents lead to a greater susceptibility to soil erosion (Wischmeier and Smith, 1978; Chaney and Swift, 1984; Bonilla and Johnson, 2012 that more silt and less organic matter in the soil enhance erodibility (Bonilla & Johnson, 2012; Chaney & Swift, 1984; Meyer & Harmon, 1984; Wischmeier & Smith, 1978). Adding polyester microplastic MP fibers had no effect on soil erosion in the lessleast erodible soil (Vertisol), whereas it resulted in pronounced reductions in the most erodible soil (a pronounced decrease of soil erosion in the more erodible soils (Entisol, Alfisol). In other words, the tendency of microplastics to decrease soil erosion increased as the soil became intrinsically more erodible. The data therefore showshowed that contamination with polyester microplastic fibers did not modify MP fibers modified only a little and not for all soils the soil hydrological behaviour (rainfall partition into surface runoff and percolation), but it strongly affected affected the soil erosion phenomena. This influence was moderate or even negligible when the soil was inherently less erodible as a consequence of a relatively low silt content and a relatively high soil organic matter content Another way to summarise these results is that a decrease of soil erosion (Fig. 8) was linked with a slight decrease of the total runoff (Fig. 7). The MP influence on soil erosion was moderate or even negligible when the soil was inherently little erodible. Instead, it became appreciable in those cases in which the erosion phenomena were noticeable since there was more silt and less organic matter. The anti-erosive effect of microplastics was more relevant as silt increased and organic matter decreased. This interpretation presupposes that the addition of microplastics essentially made the bonds among particles stronger and the Sinder values supported this conclusion. In other terms, adding microplastics made the



**Figure 9**. Experimental units of Experiment 2 (Vertisol left hand side and Alfisol right hand side) after the rainfall simulation.

soil structurally more stable, particularly when the soil was not inherently very stable. Therefore, this investigation suggested that the addition of microplastics did not alter appreciably its response in terms of surface runoff and percolation, but it modified the susceptibility of the soil particles to be detached by rainfall and surface runoff.

## 5 Conclusion.

At the end of the rainfall experiment, we observed differences between the Ctr and MP trays at the soil surface. In the trays of the Ctr treatment, micro-rills oriented parallel to the slope appeared during the rainfall-runoff event, especially in the contact zone between the soil and the box walls, or small cracks developed soon after rainfall, depending on the soil type (Fig. 9). Instead, neither micro-rills nor cracks were ever detected in the soil trays of the MP treatment. Therefore, it seems that the polyester MP fibers performed a soil particle binding action, possibly microbially-mediated, that likely induced a decrease of soil erodibility. This decrease was not suggested by the sieving experiments, probably because mechanical impact of sieving cannot be considered equivalent to the impact of the raindrops (Fox & Le Bissonnais, 1998; Loch, 1994).

Moreover, after rainfall, the soil surface of the Ctr trays generally appeared smoother than the ones of the MP trays and, especially in the darker soil, the polyester MP fibers were noted to form a diffuse fluff on the soil surface by the end of the experiment (Fig.9). Therefore, polyester MP fibers appeared to generally induce a greater resistance of the soil to flow tractive forces. An additional possible reason for the observed results was that these fibers were exposed to some degree as a consequence of erosion during the early stage of the experiment but they remained entrapped by the subsoil. Therefore, the soil surface of the MP trays appeared to have an additional micro-roughness compared to that of the Ctr trays. Consequently, flow velocity and sediment transport capacity were likely greater in the Ctr trays than in the MP ones (Zhang et al., 2011). Perhaps, the fluff formed by the exposed fibers also contributed to limit rainfall detachment, acting as a mulch.

The active erosion processes vary with the measurement scale (Cammeraat, 2002). At the soil trays scale adopted in this investigation, erosion is expected to be due to rainfall impact and interrill flow (Bagarello & Ferro, 2004) and it should be a transport-limited process as a consequence of the reduced rain impacted flow and the limitation of flow velocity (Boix-Fayos et al., 2006; Chaplot & Le Bissonnais, 2000). Therefore, the collected data provide information on particle detachment and the early stage of their transport that can be expected to occur in upland agricultural soils during intense rainfall events. However, in agricultural fields, rill erosion can dominate total soil erosion due to the simultaneous occurrence of long slopes (dozens of meters or more) and the exposure of bare soil surfaces to rainfall in some periods of the year (Rejman & Brodowski, 2005). The effects of polyester MP fibers on rill erosion require specific testing but, also according to this investigation, it could be expected that rill development is hindered since the presence of the contaminant makes the soil intrinsically less erodible. Additional investigations should also be carried out by varying the steepness of the soil trays to verify if a linear or less than linear relationship between interrill erosion rate and slope (Fox & Bryan, 2000) remains detectable after MP fibers addition into the soil.

## **5** Conclusion

Although the current MP contamination level in agroecosystems is some orders of magnitude below the concentration applied in our experiment, in some areas, it is steadily increasing (Büks & Kaupenjohann, 2020); therefore it is of key importance to investigate such contamination levels which may represent future scenarios, as is common practice in global change biology. The results showed that the presence of polyester <u>microfibersMP fibers</u> limits the neoformation of soil aggregates; <u>likely, the</u> <u>microplastic fibers hinder the natural aggregation processes and, due to their shape and size and interposing (labile in</u> incubation period applied in our experiments). Likely, polyester MP fibers hinder the natural aggregation processes since they interpose between the fine soil particles, and hence reduce the possibility of establishing bonds between them. Our data also highlighted how polyester microplastic fibers increase the water stability of aggregates; this result is somewhat unexpected, as it seems logical to assume these. Such results seem in contrast with what we observed in our rainfall simulation experiment where polyester MP fibers reduced soil loss and sediment concentration, especially in the most erodible soils, which would suggest that the polyester microplastic MP fibers, by weakening the bonds and representing fracture lines within the aggregates, reduce their stability in water. Probably these negative effects were counterbalanced by a physical protective action offered by the fibers, which, by intercepting the raindrops, allowed the dissipation of their kinetic energy and limited the splash effect that leads to the disintegration of the aggregates. This may explain why, in some cases, MP soil contamination has resulted in a very marked reduction in water erosion.

Finally, it emerged had a soil particle binding effect that reduced erodibility of the porous medium. In particular, the lack of rills in the MP treated trays suggested that polyester MP fibers induced a greater resistance of the soil to flow tractive forces of runoff. Other factors that could have played a role on the observed results include: i) mechanical impact of sieving cannot be considered equivalent to raindrop impact; ii) the presence of polyester MP fibers favoured a higher micro-roughness at the soil surface as compared with the non-treated soil and consequently the flow velocity and sediment transport capacity decreased with the addition of polyester MP fibers; and iii) the contaminant, acting as a mulch, could have exerted a physical protective action by intercepting the raindrops, allowing the dissipation of their kinetic energy, and limiting the splash effect that leads to the disintegration of the aggregates.

In conclusion, our experiments showed that the contamination of soils with <u>MPpolyester MP fibers</u> exerts an impact on the <u>soil</u> structure, susceptibility to erosion and other hydrological characteristics of the soil as a function of soil type. This aspect is of great interest and underlines how a complete understanding of the potential impacts of <u>MPpolyester MP fibers</u> on terrestrial ecosystems requires special attention to the processes that occur in the soil and to the knowledge of the mechanisms underlying the different responses.

The applied experimental methodologies in this investigation provide much information on the effect of <u>MPpolyester MP</u> <u>fibers</u> addition on several physical soil properties and hydrological processes. These methodologies should be applied in the future to different <u>MPMPs</u> concentrations (and types) to better understand the effects of <u>MPthose contaminants</u> on different soils and to help cover the potentially very large parameter space that, as suggested by <u>Rillig Rillig and Lehman& Lehmann</u> (2020) (2020), represents a major challenge of <u>MPMPs</u> research in terrestrial ecosystems.

### Author contributions

All authors (RI, GA, VB, FGC, DG, MI, AL, MCR, and ASF) conceptualized the study. GA, DG, and ASF acquired the funds to conduct the experiment. RI, FGC, DG, MI, and ASF carried out the experiments. RI, DG, and ASF analyzed the data.–\_RI, DG, GA, ASF, MI, and VB wrote the first draft of the manuscript. AL and MCR collaborated on the ideas and contributed critically to the drafts. All authors gave the final approval for publication.

## **Competing interests**

The authors declare that they have no conflict of interest.

## Acknowledgments

The authors acknowledge funding from the University of Palermo (Palermo, Italy).

## References

Alimi<u>Alimi</u>-O.S., O. S., Farner Budarz, Farner Budarz, J., Hernandez J., Hernandez L.M., Tufenkji N. (2018). Microplastics and nanoplastics in aquatic environments: aggregation, deposition, and enhanced contaminant transport, L. M., & Tufenkji, N. (2018). Microplastics and Nanoplastics in Aquatic Environments: Aggregation, Deposition, and Enhanced Contaminant Transport. *Environmental Science & Technology*, *52*(4). *Environmental Science & Technology*, *52*(4): 1704-, 1704-1724. https://doi.org/10.1007/s13369-020-04402-z10.1021/acs.est.7b05559

Bagarello, V., & Ferro, V. (2004). Plot-scale measurement of soil erosion at the experimental area of Sparacia (southern Italy). *Hydrological Processes*, *18*(1), 141–157. https://doi.org/10.1002/hyp.1318

- Bagarello, V., Ferro, V., Ferro, V., Flanagan D.C. 2018 & Flanagan, D. (2018). Predicting plot soil loss by empirical and process oriented approaches. A review. *Journal of Agricultural Engineering*, Predicting plot soil loss by empirical and process-oriented approaches. A review. *Journal of Agricultural Engineering*, vol. XLIX:710, 1–18, DOI: 10.4081/jae.2018.71049(1), 1–18.
- Bergami, E., Rota, E., Caruso, T., Birarda, G., Vaceari, L., Bergami, E., Rota, E., Caruso, T., Birarda, G., Vaccari, L., & Corsi, I. (2020). Plastics everywhere: <u>firstFirst-evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus</u>. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarctic collembolan Cryptopygus antarcticus. *Biology* evidence of polystyrene fragments inside the common Antarcticus evidence of polystyrene fragments evidence of polystyrene fragments evidence evidence
- Bonilla C.A., Johnson O.I. 2012. Soil erodibility mapping and its correlation with soil properties in Central Chile. Geoderma, 189-190: 116-123 https://doi.org/10.1098/rsbl.2020.0093
- Boix-Fayos, C., Martínez-Mena, M., Arnau-Rosalén, E., Calvo-Cases, A., Castillo, V., & Albaladejo, J. (2006). Measuring soil erosion by field plots: Understanding the sources of variation. *Earth-Science Reviews*, 3–4(78), 267–285https://doi.org/10.1016/j.geoderma.2012.05.005j.earscirev.2006.05.005
- Boots, B., Russell B., Russell C.W., Green D.S. (2019). Effects of microplastics in soil ecosystems: above and below ground, C. W., & Green, D. S. (2019). Effects of Microplastics in Soil Ecosystems: Above and Below Ground-*Environmental Science & Technology*, 53(19). Environmental Science & Technology, 53(19): 11496–, 11406–, 1140–, 1140–, 1140–, 1140–, 11400–, 1140–, 1140–,
- Bradford, J. M., Ferris, J. E., & Remley, P. A. (1987). Interrill Soil Erosion Processes: II. Relationship of Splash Detachment to Soil Properties. Soil Science Society of America Journal, 51(6), 1571–1575.
   https://doi.org/10.2136/sssaj1987.03615995005100060030xBradford, J. M., Ferris, J. E., & Remley, P. A. (1987). Interrill Soil Erosion Processes: II. Relationship of Splash Detachment to Soil Properties. Soil Science Society of America Journal, 51(6), 1571–1575.
   bit provide the state of the s
- Christiansen J.E. 1942. Irrigation by Sprinkling. California Agricultural Experiment Station Bulletin 670.
- Crossman J., Hurley R.R., Futter M., Nizzetto L. (2020). Transfer and transport of microplastics from biosolids to agricultural soils and the wider environment. Science of the Total Environment, 724, 138334.
- Dane J.H., Hopmans J.W. 2002a 3.3.2.2 Hanging water column. p.680-683. In J.H.Dane and G.C.Topp (co-eds.), Methods of Soil Analysis, Part 4, Physical Methods, Number 5 in the Soil Science Society of America Book Series, Soil Science Society of America, Inc., Madison, WI, USA.
- Dane J.H., Hopmans J.W. 2002b 3.3.2.4 Pressure plate extractor. p.688–690. In J.H.Dane and G.C.Topp (co-eds.), Methods of Soil Analysis, Part 4, Physical Methods, Number 5 in the Soil Science Society of America Book Series, Soil Science Society of America, Inc., Madison, WI, USA.
- de Souza Machado, A. A., Lau, C. W., Kloas, W., Bergmann, J., Bachelier, J. B., Faltin, E., ... & Rillig, M. C. (2019). Microplastics can change soil properties and affect plant performance. Environmental science & technology, 53(10), 6044-6052. https://doi.org/10.1021/acs.est.9b01339

- de Souza Machado, A.A., Lau, C.W., Till, J., Kloas, W., Lehmann, A., Beeker, R., Rillig, M.C., 2018. Impacts of microplastics on the soil biophysical environment. Environ. Sci. Technol. 52 (17), 9656–9665Büks, F., & Kaupenjohann, M. (2020). Global concentrations of microplastics in soils – a review. SOIL, 6(2), 649–662. https://doi.org/10.5194/soil-6-649-2020
- Cammeraat, L. H. (2002). A review of two strongly contrasting geomorphological systems within the context of scale. *Earth* Surface Processes and Landforms, 27(11), 1201–1222. https://doi.org/10.1002/esp.421
- Chaplot, V., & Le Bissonnais, Y. (2000). Field measurements of interrill erosion under -different slopes and plot sizes. *Earth* Surface Processes and Landforms, 25(2), 145–153. https://doi.org/10.1002/(SICI)1096-9837(200002)25:2<145::AID-ESP51>3.0.CO;2-3

Christiansen, J. E. (1942). Irrigation by sprinkling (Vol. 4). University of California Berkeley.

- Crossman, J., Hurley, R. R., Futter, M., & Nizzetto, L. (2020). Transfer and transport of microplastics from biosolids to agricultural soils and the wider environment. *Science of The Total Environment*, *724*, 138334. https://doi.org/10.1016/j.scitotenv.2020.138334
- Dane, J. H., Hopmans, J. W., & Topp, G. C. (2002a). Hanging water column. Methods of Soil Analysis. Part, 4, 680-683.
- Dane, J. H., Hopmans, J. W., & Topp, G. C. (2002b). Pressure plate extractor. Methods of Soil Analysis. Part, 4, 688-690.
- Deviren Saygın, S., Cornelis, W. M., Erpul, G., & Gabriels, D. (2012). Comparison of different aggregate stability approaches for loamy sand soils. *Applied Soil Ecology*, *54*, 1–6. https://doi.org/. https://doi.org/10.1021/acs.est.8b02212.10.1016/j.apsoil.2011.11.012
- Dris, R., Gasperi, R., Gasperi, J., J., Tassin & Tassin, B. (2018). Sources and B. (2018). Sources and fate of microplastics in urban areas: a focus Fate of Microplastics in Urban Areas: A Focus on Paris on Paris megacityMegacity. In. In Freshwater Microplastics M. Wagner & S. Lambert (Eds.), Freshwater Microplastics: Emerging Environmental Contaminants? (pp. 69–83). Springer, Cham.
- Gee G.W., Bauder J.W., Klute A. (1986). Methods of soil analysis, part 1, physical and mineralogical methods. Soil Science Society of America Book Series. American Society of Agronomy, Inc. and Soil Science Society of America, Inc. Madison, Wisconsin, 404-410. International Publishing. https://doi.org/10.1007/978-3-319-61615-5\_4
- Fox, D. M., & Le Bissonnais, Y. (1998). Process-Based Analysis of Aggregate Stability Effects on Sealing, Infiltration, and Interrill Erosion. *Soil Science Society of America Journal*, 62(3), 717–724. https://doi.org/10.2136/sssaj1998.03615995006200030025x
- Fox, D. M., & Bryan, R. B. (2000). The relationship of soil loss by interrill erosion to slope gradient. Catena, 38(3), 211-222.
- Gee, G. W., & Bauder, J. W. (1986). Particle-size Analysis. In *Methods of Soil Analysis* (pp. 383–411). John Wiley & Sons, Ltd. https://doi.org/10.2136/sssabookser5.1.2ed.c15
- Hartmann, N. B., Hartmann, N. B., HufferHüffer, T., Thompson, R. C., T., Thompson, R. C., HassellövHassellöv, M., Verschoor, A., Daugaard, A. E., Massellöv, M., Verschoor, A., Daugaard, A. E., M., Verschoor, A., Daugaard, A. E., Massellöv, S., Karlsson, T., Brennholt, N., Cole, M., Herrling, M. P., Hess, M. C., Ivleva, N. P., Lusher, A. L., & Wagner, M. (2019). Are We Speaking the Same Language? Recommendations for a Definition and Categorization Framework for Plastic Debris. *Environmental Science & Technology*, 53(3), 1039–1047. https://doi.org/10.1021/acs.est.8b05297
- Ho<u>Ho</u>, J<u>J</u>, Tumkaya, Tumkaya, T<u>T</u>, Aryal, Aryal, S<u>S</u>, Choi, Choi, H<u>H</u>, & Claridge-Chang Claridge-Chang, A. <u>A.</u> (2019). Moving beyond P values: <u>Moving beyond P values</u>: <u>dataData analysis with estimation graphics</u>. <u>analysis with estimation</u> graphics. <u>Nat Methods</u>. 2019 Jul;16(7):565–6.

Horton A.A.Nature Methods, 16(7), 565-566. https://doi.org/10.1038/s41592-019-0470-3

- Horton, A. A., Walton, Malton, A., Spurgeon A., Spurgeon-D.J, D. J., Lahive, Lahive, E., E., Svendsen & Svendsen, C. (2017). Microplastics in freshwater and terrestrial environments: C. (2017). Microplastics in freshwater and terrestrial environments: evaluatingEvaluating the current understanding to identify the knowledge gaps and future research priorities. Science of the current understanding to identify the knowledge gaps and future research priorities. Science of the Internet. 586 Total Environment, 586: 127–141, 127–141. https://doi.org/10.1016/j.scitotenv.2017.01.190
- Ingraffia, R., Amato, G., Iovino, M., Rillig, M. C., Giambalvo, D., & Frenda, A. S. (2022). Polyester microplastic fibers in soil increase nitrogen loss via leaching and decrease plant biomass production and N uptake. *Submitted*<sub>7</sub>.
- Iovino, M., Castellini, M., Bagarello, V., Iovino, M., Castellini, M., Bagarello, V., & Giordano, G., Giordano, G., 2016
   (2016). Using Static and Dynamic Indicators to Evaluate Soil Physical Quality in a Sicilian Area. Land Degradation & Development. Using Static and Dynamic Indicators to Evaluate Soil Physical Quality in a Sicilian Area. Land Degradation & Development, 27(2), 200 27(2), 200-210. https://doi.org/10.1002/ldr.2263
- Iserloh, T., Fister, T., Fister, W., Seeger, M., Willger, M., Willger, H., H., Ries J.B& Ries, J. B. (2012). A small portable rainfall simulator for reproducible experiments on soil erosion. *Soil and Tillage Research*, *124*. (2012). A small portable rainfall simulator for reproducible experiments on soil erosion. *Soil and Tillage Research*, *124*. (2012). A small portable rainfall simulator for reproducible experiments on soil erosion. *Soil and Tillage Research*, *124*. (2012). A <u>137. URL https://doi.org/10.1016/j.still.2012.05.016</u>https://doi.org/10.1016/j.still.2012.05.016-
- Iserloh, T., Ries T., Ries J.B, J. B., Arnáez, Arnáez, J., Boix Fayos J., Boix-Fayos, C., Butzen, C., Butzen, V., Cerdà
   V., Cerdà, A., Echeverría A., Echeverría M.T, M. T., Fernández Gálvez, Fernández-Gálvez, J., Fister J., Fister, W., W., Geißler C., Gómez J.A., Gómez Macpherson H., Kuhn N.J., Lázaro R., León F.J., Martínez Mena M., Martínez Murillo
   J.F., Marzen M., Mingoranee M.D., Ortigosa L., Peters P., Regüés D., Ruiz-Sinoga J.D., Scholten T., Seeger M., Solé-Benet A., Wengel R., Wirtz S. 2013. European small portable rainfall simulators: A comparison of rainfall characteristics. Catena, 110: 100–112, https://doi.org/10.1016/j.catena.2013.05.013.& Geißler, C. (2013). European small portable rainfall simulators: A comparison of rainfall characteristics. Catena, 110, 100–112.

Lal, R., & Shukla, M. K. (2004). Principles of Soil Physics. CRC Press. https://doi.org/10.4324/9780203021231

- Lehmann, A., Fitschen, K., & Rillig, M. C. (2019). Abiotic and Biotic Factors Influencing the Effect of Microplastic on Soil Aggregation. Soil Systems, 3(1), 21. https://doi.org/10.3390/soilsystems3010021
- Lehmann, A., Leifheit A., Leifheit E.F, E. F., Feng., Feng., Feng., L., Bergmann, L., Bergmann, J., Wulf, J., Wulf, A., A., Rillig M.C. (2020). Microplastic Fiber and Drought Effects on Plants and Soil Are Only Slightly Modified by Arbuscular Mycorrhizal Fungi. Soil Ecol. Lett. <u>https://doi.org/10.1007/s42832-020-0060-4</u>.
- Liang, Y., Lehmann, A., Yang, G., Leifheit, E. F., & Rillig, M. C. (2021). Effects of Microplastic Fibers on Soil Aggregation and Enzyme Activities Are Organic Matter Dependent. Frontiers in Environmental Science, 9, 97.
- Liu M., Lu S., Song Y., Lei L., Hu J., Lv W., Zhou W., Cao C., Shi H., Yang X., He D. (2018). Microplastic and mesoplastic pollution in farmland soils in suburbs of Shanghai, China. Environmental Pollution, 242, 855-862.
- Lozano Y.M& Rillig, M. C. (2020). Microplastic fiber and drought effects on plants and soil are only slightly modified by arbuscular mycorrhizal fungi. *Soil Ecology Letters*. https://doi.org/10.1007/s42832-020-0060-4
- Lehmann, A., Leifheit, E. F., Gerdawischke, M., & Rillig, M. C. (2021). Microplastics have shape- and polymer-dependent effects on soil aggregation and organic matter loss – an experimental and meta-analytical approach. *Microplastics and* <u>Nanoplastics</u>, 1(1), 7. https://doi.org/10.1186/s43591-021-00007-x
- Liang, Y., Lehmann, A., Yang, G., Leifheit, E. F., & Rillig, M. C. (2021). Effects of Microplastic Fibers on Soil Aggregation and Enzyme Activities Are Organic Matter Dependent. *Frontiers in Environmental Science*, 9, 97.

https://doi.org/10.3389/fenvs.2021.650155

- Loch, R. J. (1994). A method for measuring aggregate water stability of dryland soils with relevance to surface seal development. *Soil Research*, *32*(4), 687–700. https://doi.org/10.1071/sr9940687
- Lowery, B., Swan, J., Schumacher, T., & Jones, A. (1995). Physical properties of selected soils by erosion class. *Journal of* Soil and Water Conservation, 50(3), 306–311.
- Lozano, Y. M., Lehnert, Lehnert, T., Linck T., Linck L.T, L. T., Lehmann, A., A., & Rillig, Rillig, M.CM. C. (2021). Microplastic Shape, Polymer Type, and Concentration Affect Soil Properties and Plant Biomass. *Frontiers in Plant Science*, 12. (2021). Microplastic Shape, Polymer Type, and Concentration Affect Soil Properties and Plant Biomass. *Frontiers in Plant Science*, 12. (2021). Microplastic Shape, Polymer Type, and Concentration Affect Soil Properties and Plant Biomass. *Frontiers in Plant Science*, 12, 169.
- Lozano Y.M., Rillig M.C. (2020). Effects of Microplastic Fibers and Drought on Plant Communities. Environ. Sci. Technol., 54 (10), 6166–6173. https://doi.org/10.1021/acs.est.0c01051.
- Lowery, B., Swan, J., Schumacher, T., & Jones, A. (1995). Physical properties of selected soils by erosion class. Journal of Soil and Water Conservation, 50(3), 306-311.
- Maaß S., Daphi D., Lehmann A., Rillig M.C. (2017). Transport of microplastics by two collembolan species. Environmental Pollution, 225: 456-459. https://doi.org/10.3389/fpls.2021.616645
- Maaß, S., Daphi, D., Lehmann, A., & Rillig, M. C. (2017). Transport of microplastics by two collembolan species. *Environmental Pollution*, 225, 456–459. https://doi.org/10.1016/j.envpol.2017.03.009
- Machado, A. A. de S., Lau, C. W., Till, J., Kloas, W., Lehmann, A., Becker, R., & Rillig, M. C. (2018). Impacts of <u>Microplastics on the Soil Biophysical Environment. *Environmental Science & Technology*. <u>https://doi.org/10.1021/acs.est.8b02212</u></u>
- Machado, A. A., Lau, C. W., Kloas, W., Bergmann, J., Bachelier, J. B., Faltin, E., Becker, R., Görlich, A. S., & Rillig, M. C. (2019). Microplastics Can Change Soil Properties and Affect Plant Performance. *Environmental Science & Technology*, 53(10), 6044–6052. https://doi.org/10.1021/acs.est.9b01339
- Mamedov, A. I., & Levy, G. J. (2019). Soil erosion runoff relations on cultivated land: Insights from laboratory studies. *European Journal of Soil Science*, 70(3), 686–696. https://doi.org/10.1111/ejss.12759Mamedov, A. I., & Levy, G. J. (2019). Soil erosion-runoff relations on cultivated land: Insights from laboratory studies. *European Journal of Soil Science*, 70(3), 686–696. https://doi.org/10.1111/ejss.12759
- Napper, I. E., Davies, B. F. Napper, I. E., Davies, B. F. R., Clifford, H., Elvin, S., Koldewey, H. J., Mayewski, P. A., Clifford, H., Elvin, S., Koldewey, H. J., Mayewski, P. A., Miner, K. R., Potocki, M., Elmore, A. C., Gajurel, A. P., & Thompson, R. C. (2020). Reaching <u>while heights in plastic pollution</u> preliminary findings of <u>microplastics New Heights in Plastic Pollution</u> Preliminary Findings of Microplastics on Mount Everest. One Earth, 3(5), 621 on Mount Everest. One Earth, 3(5), 621–630.630.
- Nelson D.W., Sommers L.E. (1996). Total carbon, organic carbon, and organic matter. Methods of soil analysis: Part 3 Chemical methods, 5, 961-1010.
- Pinheiro, J., Bates, D., DebRoy, S., Sarkar, D., Team, R.C., 2018. nlme: Linear and nonlinear mixed effects models, R package version 3.1–137 ed.
- Qi R., Jones D.L., Li Z., Liu Q., Yan C. (2020). Behavior of microplastics and plastic film residues in the soil environment: A critical review. Science of the Total Environment, 703: 134722.
- Qi Y, Yang X, Pelaez AM, Lwanga EH, Beriot N, Gertsen H, Garbeva P., Geissen V. 2018. Macro-and micro-plastics in soil-plant system: effects of plastic mulch film residues on wheat (*Triticum aestivum*) growth https://doi.org/10.1016/j.oneear.2020.10.020

Nelson, D. W., & Sommers, L. E. (1996). Total Carbon, Organic Carbon, and Organic Matter. In *Methods of Soil Analysis* (pp. 961–1010). John Wiley & Sons, Ltd. https://doi.org/10.2136/sssabookser5.3.c34

O'Kelly, B. C., El-Zein, A., Liu, X., Patel, A., Fei, X., Sharma, S., Mohammad, A., Goli, V. S. N. S., Wang, J. J., Li, D., Shi, Y., Xiao, L., Kuntikana, G., Shashank, B. S., Sarris, T. S., Hanumantha Rao, B., Mohamed, A. M. O., Paleologos, E. K., Nezhad, M. M., & Singh, D. N. (2021). Microplastics in soils: An environmental geotechnics perspective. *Environmental Geotechnics*, 8(8), 586–618. https://doi.org/10.1680/jenge.20.00179

Pinheiro, J., Bates, D., DebRoy, S., Sarkar, D., & R-core. (2021). *nlme: Linear and Nonlinear Mixed Effects Models* (3.1-152) [Computer software]. https://CRAN.R-project.org/package=nlme

- Qi, R., Jones, D. L., Li, Z., Liu, Q., & Yan, C. (2020). Behavior of microplastics and plastic film residues in the soil environment: A critical review. *Science of*. *Science of* the *The Total Environment*, *Total Environment*, 645: 1048-1056. 703, 134722. https://doi.org/10.1016/j.scitotenv.2019.134722
- R Core Team 2020. <u>R</u> Core Team 2020. (n.d.). <u>R</u>: <u>A language and environment for statistical computing.</u> <u>RR: A language</u> <u>and environment for statistical computing.</u> <u>R</u>-<u>Foundation for Statistical Computing, Vienna, Austria. URL https://www.R-project.org/. (4.0.2) [Computer software].</u>
- RehmRehm, R., Zeyer, T., Schmidt, T., Schmidt, A., A., Fiener& Fiener, P. 2021(2021). Soil erosion as transport pathway of microplastic from agriculture soils to aquatic ecosystems. *Science of the Total Environment*, 795, 148774. Soil erosion as transport pathway of microplastic from agriculture soils to aquatic ecosystems. *Science of the Total Environment*, 795, 148774.
- Reynolds, W.DRejman, J., & Brodowski, R. (2005). Rill characteristics and sediment transport as a function of slope length during a storm event on loess soil. *Earth Surface Processes and Landforms*, *30*(2), 231–239. https://doi.org/10.1002/esp.1177
- <u>Reynolds, W. D., Drury, ., Drury, C.FC. F., Tan, ., Tan, C.SC. S., Fox, ., Fox, C.A., Yang, X.M., 2009C. A., & Yang, X. M.</u> (2009). Use of indicators and pore volume function characteristics to quantify soil physical quality. *Geoderma*. Use of indicators and pore volume-function characteristics to quantify soil physical quality. *Geoderma*. <u>152(3)</u> 152(3-4), 2524. 252-263.263.
- <u>RiesRies\_J.B., J. B., Seeger, Seeger, M., Iserloh, M., Iserloh, T., Wistorf, T., Wistorf, S., S., Fister & Fister, W. (2009).</u> Calibration of simulated rainfall characteristics for the study of soil erosion on agricultural land. *Soil and Tillage Research*, 106 W. (2009). Calibration of simulated rainfall characteristics for the study of soil erosion on agricultural land. *Soil and Tillage Research*, 106 W. (2009). Calibration of simulated rainfall characteristics for the study of soil erosion on agricultural land. *Soil and Tillage Research*, 106 (1)(1): 109-116. URL https://doi.org/10.1016/j.still.2009.07.005.

Rillig M.C, 109–116.

- <u>Rillig, M. C., Ingraffia, R., R., de Souza Machado A.A. (2017b). Microplastic incorporation into soil in</u> agroecosystems. Frontiers in Plant Science, 8: 1805. Machado, A. A. (2017). Microplastic Incorporation into Soil in Agroecosystems. *Frontiers in Plant Science*, 8. https://doi.org/10.3389/fpls.2017.01805
- Rillig, M. C., & Lehmann, A. (2020). Microplastic in terrestrial ecosystems. *Science*, 368(6498), 1430<u>Rillig, M. C., & Lehmann, A. (2020). Microplastic in terrestrial ecosystems. *Science*, 368(6498), 1430–1431.1431.</u>

Rillig M.C https://doi.org/10.1126/science.abb5979

<u>Rillig, M. C., Ziersch., Ziersch, L., L., & HempelHempel, S. (S. (2017a2017). Microplastic transport in soil by earthworms.</u> <u>Scientific</u>). Microplastic transport in soil by earthworms. <u>Scientific reportsReports</u>, 7(1), 7(1), 1-6. Topp, G.C.R., W.D.; Cook. F.J.; Kirby, J.M.; Carter, M.R., 1997. Physical attributes of soil quality. In: E.G.C. Gregorich, M.R. (Ed.), Soil Quality for Crop Production and Ecosystem Health. Development in Soil Science. Elsevier, New York, NY, pp. 21-58.

van Genuchten, M.T., 19801362. https://doi.org/10.1038/s41598-017-01594-7

- Topp, G. C., Reynolds, W. D., Cook, F. J., Kirby, J. M., & Carter, M. R. (1997). Physical attributes of soil quality. In *Developments in soil science* (Vol. 25, pp. 21–58). Elsevier.
- <u>van Genuchten, M. Th. (1980)</u>. A Closed form Equation for Predicting the Hydraulic Conductivity of Unsaturated Soils. Soil Science Society of America Journal, 44(5), 892. A Closed-form Equation for Predicting the Hydraulic Conductivity of Unsaturated Soils. Soil Science Society of America Journal, 44(5), 892–898.898.

Wasserstein R.L., Lazar N.A. https://doi.org/10.2136/sssaj1980.03615995004400050002x

- <u>Wasserstein, R. L., & Lazar, N. A. (2020)</u>. ASA Statement on Statistical Significance and p-Values. In. ASA Statement on Statistical Significance and p-Values. In: Gruber CW, editor. C. W. Gruber (Ed.), *The Theory of Statistics in Psychology: Applications, Use, and Misunderstandings*. *The Theory of Statistics in Psychology: Applications, Use, and* <u>Misunderstandings</u> [Internet]. Cham: Springer International Publishing; 2020 [cited 2020 Oct 16]. p(pp. 1–10. 1–10)... (Annals of Theoretical Psychology). Available from: Springer International Publishing. https://doi.org/10.1007/978-3-030-48043-1-1 https://doi.org/10.1007/978-3-030-48043-1\_1
- Weithmann, Weithmann, N., Möller N., Möller J.N., Löder M.G., J. N., Löder, M. G. J., Piehl, S., Laforsch, S., Laforsch, C., C., Freitag& Freitag, R. (2018). Organic fertilizer as a vehicle for the entry of microplastic into the environment. *Science Advances*, 4(4) R. (2018). Organic fertilizer as a vehicle for the entry of microplastic into the environment. *Science Advances*, 4(4):, eaap8060. eaap8060. https://doi.org/10.1126/sciadv.aap8060
- XuXu, B., Liu, B., Liu, F., Cryder, F., Cryder, Z., Huang, Z., Huang, D., Lu, D., Lu, Z., He, Z., He, Y., Wang, Y., Wang, H. H., Lu, Lu, Z., Brookes, Z., Brookes, P.C., P. C., Tang, Tang, C., Gan, C., Gan, J., J., Xu& Xu, J. (2020). Microplastics in the soil environment: J. (2020). Microplastics in the soil environment: occurrenceOccurrence, risks, interactions and fate, risks, interactions and fate-a – A review. Critical Reviews in Environmental Science and Technology, 50(21), 2175 review. Critical Reviews in Environmental Science and Technology, 50(21), 2175–2222.2222.

Zhang G.S., Liu Y.F https://doi.org/10.1080/10643389.2019.1694822

- Zhang, B., Yang, X., Chen, L., Chao, J., Teng, J., & Wang, Q. (2020). Microplastics in soils: A review of possible sources, analytical methods and ecological impacts. *Journal of Chemical Technology & Biotechnology*, 95(8), 2052–2068. https://doi.org/10.1002/jctb.6334
- Zhang, G. S., & Liu, Y. F. (2018). The distribution of microplastics in soil aggregate fractions in southwestern China. Science of <u>of</u>. (2018). The distribution of microplastics in soil aggregate fractions in southwestern China. Science of the The Total Environment, 642 Total Environment, 642-12-20.
- Zhang G.S., Zhang F.X., Li X.T. (2019). Effects of polyester microfibers on soil physical properties: Perception from a field and a pot experiment. Science of the total environment, 670: 1-7.

Zubris K.A.V., Richards B.K. 12-20. https://doi.org/10.1016/j.scitotenv.2018.06.004

Zhang, G. S., Zhang, F. X., & Li, X. T. (2019). Effects of polyester microfibers on soil physical properties: Perception from a field and a pot experiment. *Science of The Total Environment*, 670, 1–7. https://doi.org/10.1016/j.scitotenv.2019.03.149

Zhang, G.-H., Wang, L.-L., Tang, K.-M., Luo, R.-T., & Zhang, X. C. (2011). Effects of sediment size on transport capacity of overland flow on steep slopes. *Hydrological Sciences Journal*, 56(7), 1289–1299. https://doi.org/10.1080/02626667.2011.609172 Zubris, K. A. V., & Richards, B. K. (2005). Synthetic fibers as an indicator of land application of sludge. *Environmental*. (2005). Synthetic fibers as an indicator of land application of sludge. *Environmental* pollution, 138(2), 138(2); 201-211., 201-211. https://doi.org/10.1016/j.envpol.2005.04.013