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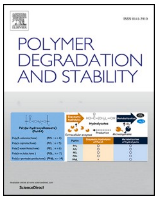

Inhibition factors and soil contamination by macro/micro-particles related to biodegradation of mulching films in Mediterranean and northern climates

Demetres Briassoulis [a,*], Christina Pyromali [a], Fabiana Convertino [b], Evelia Schettini [b], Sabrina Carola Carroccio [c], Pierfrancesco Cerruti [d], Sarmite Kernchen [e], Sandro Dattilo [c], Johanna Nikama [f], Helena Soinne [f], Giuliano Vox [b], Salla Selonen [g], Martin G.J. Löder [e], Christian Laforsch [e], Antonis Mistriotis [a]

[a] *Department of Natural Resources & Agricultural Engineering, Agricultural University of Athens, Athens, Greece*
[b] *Department of Soil, Plant and Food Sciences, University of Bari Aldo Moro, Bari, Italy*
[c] *Institute of Polymers, Composites and Biomaterials (IPCB), National Research Council (CNR), Catania, Italy*
[d] *Institute of Polymers, Composites and Biomaterials (IPCB), National Research Council (CNR), Italy*
[e] *Chair of Animal Ecology I, BayCEER, University of Bayreuth, Bayreuth, Germany*
[f] *Natural Resources Institute Finland (Luke), Helsinki, Finland*
[g] *Finnish Environment Institute (Syke), Helsinki, Finland*

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ABSTRACT

Key knowledge gaps for the biodegradability in soil of bio-based biodegradable mulching films include potential inhibition factors related to different climate zones, environmental conditions, and material composition. Possible soil pollution by macro/micro/nano-particles of the degraded mulching films has also been poorly investigated so far. These gaps, stemming from the lack of comprehensive, full-scale comparative studies, were addressed by examining the disintegration behaviour as an indication of biodegradation of two commercially available agricultural mulching films. Both films were manufactured from the same starch/PBAT-based raw material and certified as soil biodegradable. One type was tested under field conditions in southern (Greece) and northern (Finland) Europe, while the second type was tested in southern Europe (Italy). In Greece, the film fully disintegrated in 5-7 months, while in Finland, two dominant inhibiting factors suppressed its disintegration to 32% in 29 months: a) lower temperatures throughout the year ($T_{winter}<0^{\circ}C$), and b) acidic soil, impeding the microbial activity. The disintegration of the second film type in Italy, under analogous climatic conditions to Greece, was significantly lower (84% in 29 months), because of an analytically identified deviation of the film's composition. The analysis of the soils revealed a low number of micro-particles originating from the biodegradable film exposed in Greece and Finland. Macro, and mainly micro-particles originating from the mulching film exposed in Italy, were identified, confirming the inhibition effect of the material. New grades of soil biodegradable materials, designed to be functional and biodegradable under different climate zones and soil environments, should be urgently developed.

1. Introduction

Mulching films represent a dynamic category of agricultural films offering multiple benefits for various crops (e.g., one season horticultural cultivations, or perennial plants like vines, orchards, strawberries etc.). The benefits of mulching films include weed control, improvement of water conservation, and soil temperature regulation depending on the pigments used. These benefits contribute to reduced water usage, lower inputs of agrochemicals and labour, optimization of the harvesting time, higher yields and improved product quality [1,2]. The conventional fossil-based plastic mulching films are usually made of polyethylene (PE): generally mono-layer, but recently also multilayer, depending on

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* Corresponding author.
E-mail address: briassou@aua.gr (D. Briassoulis).

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the application and the required characteristics. Thin films (from 10 to 25 μm thick) are made of high strength linear low-density PE (PE-LLD), while thick one-season films (25 - 32 μm) are made of low-density PE (PE-LD), depending on the expected useful lifetime. Conventional mulching films should be certified according to the standard specification EN 13655:2018 [3].

The global market of agricultural plastics used in plant production and livestock farming in 2019 was estimated at 12.5 million tons (Mt) annually, and plastics used in food packaging at 37.3 Mt [1]. The use of agricultural plastics is expanding worldwide into new geographical regions, and new applications incorporating advances in both materials and technologies are being adopted for sustainable and precision farming. Among the various categories of agricultural plastics, agricultural films are the dominant category for plant production and livestock farming [4]. The global agricultural films market, estimated at $13.8 bn in 2024, is forecast to reach a value of $19.5 bn by 2029, growing with a compound annual growth rate (CAGR) of 7.1% [5]. The mulching films category, with the highest share in the agricultural films market and an estimated value of $7.8 bn in 2023, is predicted to reach a value of $10.6 bn in 2028, with a CAGR of 6.5% [6].

The major problem of the conventional (i.e., fossil-based, non-biodegradable) mulching films is the practical difficulty in removing the degraded films from the field at the end of the cultivation season, and the fact that these thin films degraded and contaminated by soil and organic matter are non-recyclable [7]. As a result, the used mulching films are often mismanaged (e.g., discarded in the fields, burned or buried, or abandoned in the surrounding environment), with a major environmental pollution impact, including the generation of micro/-nano- plastics [4].

A promising environmentally sustainable alternative to non-biodegradable fossil-based mulching films is the use of films certified as bio-based biodegradable in soil designed to degrade primarily by soil microbial activity. There have been major advances in recent years in the development of innovative (mostly partially bio-based) mulching films biodegradable in soil, with properties comparable to those of the conventional films, without however, their negative end-of-life (EoL) environmental impact. The thickness of the biodegradable mulching films (BDMs) is usually 15 μm to facilitate biodegradation. They are made of various blends of bio-based polymers (e.g., polyhydroxyalkanoates (PHAs), PHA/PLA etc.), or blends of bio-based and fossil-based polymers (e.g., polybutylene adipate terephthalate (PBAT) with modified starch or polylactic acid (PLA)), or blends based on polybutylene succinate (PBS) and polycaprolactone (PCL) [8–10]. PBAT and PBS are mostly fossil-based, but recently, partially bio-based PBAT and Bio-PBS polymers have been produced at industrial level, and they are now commercially available [11]. The fast growth of bio-based mulching films biodegradable in soil is reflected in the corresponding market. The global market of BDMs, estimated at $50.8 mm in 2022, is predicted to reach a value of $55.6 mm with a high CAGR of 9.5% [12].

Despite their increasing adoption, the extent and efficiency of BDMs biodegradation under field conditions is controversial. Degradation in open natural environments depends on complex interactions among the physicochemical properties of the film, the soil characteristics, and the climatic conditions. Understanding these dynamics is fundamental for evaluating the performance of BDMs in real-world agricultural applications [13,14]. Biodegradation of BDMs involves hydrolysis and enzymatic breakdown of the polymer matrix by microorganisms (e.g., fungi, bacteria) into smaller fragments, which are subsequently assimilated by the microorganisms as an energy source and mineralized into CO_2, water, and microbial biomass. The rate and extent of their biodegradation are influenced by the chemical structure of the film. Starch-based films, for example, typically exhibit relatively rapid degradation due to the high biodegradability of starch, whereas films incorporating synthetic biodegradable polymers such as PBAT degrade more slowly and under specific conditions [15]. On the other hand, some bio-based materials (e.g., PLA) may be biodegradable under controlled conditions (e.g., aerobic composting conditions at elevated temperatures) but not in soil.

Comparative disintegration and biodegradation in soil studies of PLA and PHA mulching films under field and laboratory conditions have shown a rather slow degradation of PLA despite increased brittleness and visual deterioration [16]. Similar results have been reported in previous open field experiments in the same experimental area and laboratory tests simulating burial conditions [17]. These results agreed with the reported mechanism of degradation of PLA in soil [18], starting with a very slow hydrolysis process which takes a long time at the low temperatures of real field conditions (in contrast to high temperature composting conditions, ~58±2°C), before commencement of any biotic activity at a second stage (still a slow process). In comparison to PLA, the biodegradation of the PHA based film was much faster both under real field and controlled laboratory conditions. The biotic activity starting from the beginning of the soil burial exposure independently of the soil temperature agreed with the layer-by-layer biodegradation process of PHA films proposed by Corrêa et al. [19].

Environmental factors play a critical role in the biodegradation of mulching films. Soil temperature and water content (WC) are the primary drivers, as they regulate microbial activity and enzymatic processes [20]. Warmer climates, such as in Southern Europe, often promote faster biodegradation due to prolonged ultraviolet (UV) radiation during use phase as well as the elevated microbial metabolism, provided that sufficient soil moisture is available. In a study conducted in Japan, including polymers polyhydroxybutyrate (PHB) and poly (3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), higher biodegradation rates were observed in the following order: PBSA = PHB/V = PCL > PBS > PLA [21]. Temperature over 10°C was correlated with the disintegration of these polymers. However, prolonged periods of drought, common in these regions, in the absence of irrigation, might significantly reduce microbial activity, hindering the degradation process [22]. In contrast, cooler climates may slow the microbial activity and the biodegradation rate over time. In fact a degree of disintegration due to biodegradation in soil of 61-83% for PBAT/Starch, PBAT/PLA, and PLA/PHA based BDMs, was reported at warm climate (Knoxville, Tennessee, USA) whereas the degree of disintegration was 26-63% in cooler climate (Mount Vernon, Washington, USA) during a period of 36 months [23]. Soil properties, including organic matter content, pH, and microbial diversity, further influence biodegradation rates. Hoshino et al. [24], reported total nitrogen in soil to be correlated with biodegradation at various times of exposure, while total carbon, pH, and soil texture had no significant effect. Soils rich in organic matter and with diverse microbial communities are generally more conducive to BDM biodegradation. On the contrary, soils with limited microbial biomass, low nutrient availability or adverse conditions, such as low or high pH or high concentration of soluble salts, may hinder the process, raising questions about the universal applicability of BDMs across different agricultural systems [25]. Accordingly, it has been shown that the biodegradation behaviour of the same biodegradable material (e.g. reference film made of cellulose) depends on several environmental parameters, including the soil characteristics [25]. Additionally, climatic and seasonal conditions also affect the rate of biodegradation of a given material [23].

Within Europe the climatic conditions vary from northern cold climatic conditions to the southern warm and dry. Despite the potential environmental benefits of BDMs, concerns have been raised regarding their actual biodegradation in soil. Several studies have highlighted the risk of incomplete biodegradation under field conditions, particularly for specific materials, leading possibly to accumulation of macro/micro/nano-particle residues that prevent their complete mineralization in soil. These findings challenge the assumption that all BDMs are inherently environmentally harmless [11,26]. The results of a life cycle assessment (LCA) study under Nordic conditions suggested that BDMs will reach a “dynamic equilibrium” in soils between a used film biodegradation and a new film application, if not enough time is given for the used films to be fully biodegraded (i.e., several years' interval). However, the reliability of these LCA results were questioned in a case study in Norway due to “limited data availability” [27].

The in-situ biodegradation of BDMs under a large variation of different open field natural conditions and for long durations remains uncertain [28]. Studies have reported a substantial variability in BDM degradation rates between laboratory and field conditions. Laboratory tests, which often provide optimal moisture, temperature, and microbial activity, typically show faster degradation rates. Conversely, field conditions introduce environmental variability, such as fluctuations in temperature and soil moisture that can inhibit biodegradation [29]. These findings underscore the need for field-specific assessment to evaluate BDM performance accurately. Moreover, the lack of standardized methods for assessing BDM biodegradation under field conditions further complicates evaluations. The variability in the biodegradation behaviour of BDMs in the open field, however, does not allow for reliable prediction by means of standard test methods. Existing field studies are often limited in geographic scope, failing to capture the influence of diverse soil types, climates and farming practices. Addressing the gaps requires a comprehensive approach that integrates pre-standardization field trials with continuous environmental monitoring [13,15,22,30].

Along with field experiments, laboratory testing is still needed. To ensure high repeatability in comparing the biodegradability of different BDMs, which vary in material composition (polymers, additives, processing), their biodegradability must be tested under standardized laboratory conditions. This important need is covered by international standard test methods and specifications (EN 17033:2018 or ISO 23517:2021) [31,32]. According to EN 17033, BDMs are defined as biodegradable in soil based on several criteria: a) biodegradation in soil $>$90%, i.e. conversion of organic carbon into CO_2, in 2 years at ambient conditions (20-28°C, optm 25°C), ISO 17556:2019 [33]; b) chemical composition (regulated metals and hazardous substances); c) ecotoxicity (plants, invertebrates, microorganisms); d) physical characteristics. BDMs can be certified according to the standard specifications (EN 17033:2018 or ISO 23517:2021) [32,33]. However, available accredited certification and/or labelling schemes for biodegradable in soil mulching films are limited (e.g., certification DIN CERTCO (EN 17033), TUV Austria (ISO 17556, ISO 11266, ASTM D5988)) [34]. It is noted that the standards, such as a European Norm, are voluntary guidelines that provide technical specifications for certain goods, services and processes. Their use is very important for the market. It is also important to note that standard laboratory test methods and specifications for biodegradability in soil are not designed to simulate field dynamics across different climatic conditions. Instead, they provide a reliable and repeatable basis for comparing different BDMs under controlled conditions. Field studies, however, are needed to verify the biodegradability across various climatic zones and soil types.

Concerning the terminology used and relevant definitions: *“The ‘microplastic’ term definition is not consistent or standardized. According to European Chemicals Agency (ECHA), the term ‘microplastic’ typically refers to small, usually microscopic, solid particles made of a synthetic polymer. They are associated with long-term persistence in the environment, if released, as they are very resistant to (bio)degradation”*[4]. This research concerns both persistent macro/micro-plastics originating from various indirect sources of conventional non-degradable plastics as well as biodegradable macro/micro-particles originating from certified soil biodegradable mulching films of gradually diminishing size. To avoid complications with two different abbreviations the authors are adopting the abbreviation term MP for both groups: the term MP is used for both macro/micro-plastics (MP) in the case of non-degradable, persistent in soil macro/micro-plastics, and macro/micro-particles (MP) in the case of macro/micro-particles from soil biodegradable mulching films, of gradually diminishing size until full biodegradation.

Scope of the work: This study addresses key knowledge gaps for the biodegradability in soil of bio-based certified soil biodegradable mulching films, including possible inhibition factors related to different climate zones, environmental conditions, and composition of the materials. Possible soil pollution by MP of biodegraded mulching films is largely unknown. These gaps, due to limited comparative full-scale studies, were investigated through the comparative disintegration, as an indication of biodegradation, of mulching films biodegradable in soil under field conditions in Southern and Northern Europe. By identifying the dominant inhibition factors and the related soil contamination by MP, this research aimed to provide a comprehensive evaluation of BDMs performance across different geographical locations. The findings are expected to contribute to better understanding the different behaviour of BDMs in different soil environments, the development of improved biodegradable materials, reliably certified and applied, and propose policy strategies for their effective implementation in diverse agricultural systems without negative effects for the environment, to advance sustainable agriculture on a global scale. It is noted that the analysis of MPs' impacts on soil was beyond the scope of this work.

2. Materials and methods

2.1. Test materials

Two different types of commercial agricultural biodegradable mulching films, 15 μm thick, were selected for the field experiments: BIOEL film was manufactured in Greece by a Greek industry. It was supplied directly to the Agricultural University of Athens (AUA) for use in the field experiments in Greece and Finland. BIOIT was manufactured by an Italian industry. It was supplied directly to the University of Bari (UNIBA) for use in the field experiment of Italy. According to the manufacturers, both films were composed of the same raw material supplied by an Italian industry made of a blend of modified starch and PBAT (Table 1). The films also contained carbon black (CB) (using different masterbatches). BIOEL, certified as “OK Biodegradable Soil” by TUV-Austria, contained 1.4% premium P-type CB with an average particle size 20-25 nm, carried in a PBAT resin-based masterbatch, with a CB concentration of 35%. The concentration of the PBAT resin-based CB masterbatch in the film was 4%. The film BIOIT was certified as “Biodegradable in soil (2018-03) according to DIN EN 17033:2018-03” by DIN-CERTCO. It contained about 3 wt% type P-black CB pigment. This film was prepared using a PBAT-based masterbatch containing about 40 wt% CB. No information has been disclosed by the manufacturers about the possible use of other additives.

2.2. Material characterization methods

2.2.1. Artificial ageing

Artificial ageing of the tested BDMs samples under controlled laboratory conditions was conducted at the Agricultural University of Athens (AUA, Greece) and at the Institute of Polymers, Composites and Biomaterials - National Research Council (CNR-IPCB, Catania Unit, Italy) to simulate the photodegradation of the mulching films during their exposure to solar radiation under open field cultivation conditions. The photodegradation of the mulching films speeds up the biodegradation process of their residues that remain in the field and are rototilled in the soil after the crop cycle's end. The artificially aged biodegradable mulching films simulate the film residues degraded by solar radiation.

Pieces of the pristine mulch film BIOEL were exposed at AUA to UV-A radiation in specially designed chambers equipped with fifteen UVA340 lamps (40 W). The total UV-A irradiation on the film surface was maintained at 38.5±1 W/m^2 measured by a CUV3 Kipp & Zonen UV-radiometer. The spectrum of the UVA340 lamps (Q-Lab Corporation, Westlake, OH, USA) with a maximum spectral radiance at the wavelength of 340 nm simulates the solar UV radiation spectrum. The temperature in the chambers during film exposure was controlled and maintained at 60°C. The ageing process at AUA was closely monitored by collecting samples from the BIOEL film every day and measuring the

Table 1
Characteristics of the two biodegradable mulching films.

Material	Thickness (μm)	Composition	Additives (%) Masterbatch (%)	Tensile strength MD/TD (MPa)	Elongation at break MD/TD (%)	Certifications
BIOEL	15	Starch/PBAT blend	CB in the film: 1.4 CB in PBAT-based masterbatch: 35.0	29.30 ± 1.70/ 28.50 ± 3.70	363.80 ± 35.10/ 567.20 ± 43.30	Raw material: "OK Biodegradable Soil" (TUV Austria) Film: "OK Biodegradable Soil" (TUV)
BIOIT	15	Starch/PBAT blend	CB in the film: ~ 3.0 CB in PBAT-based masterbatch: 40.0	26.1 ± 1.95/ 16.13 ± 1.73	300.05 ± 7.85/ 207.65 ± 6.81	Raw material: "OK Biodegradable Soil" (TUV Austria) Film: "Biodegradable in Soil DIN EN 17033:2018-03" (DIN-CERTCO)

mechanical properties and the chemical structure of the samples. The exposed film samples were considered degraded and removed from the chamber when their elongation at break value (ε_{br}) was reduced to 50% of the original ε_{br} value.

A similar photo-oxidative degradation experiment of the mulching film was carried out at CNR-IPCB. The mulch film BIOIT was exposed to accelerated weathering tests in a Q-UV Panel apparatus at 60°C (Q-Labs Corp., Westlake, OH, USA) containing eight UVA340 lamps with maximum irradiation at 340 nm. The spectral irradiance at the spectrum peak was set at 0.68 W/m^2/nm resulting to total UV irradiation on the film surface equal to 39.2 W/m^2. Film specimens were irradiated for 192 h, which corresponds to 850 MJ/m^2 of exposure to solar radiation in an open field, i.e., approximately two spring months in Southern Italy [35].

Both accelerated weathering tests were carried out using continuous UV radiation, in the absence of water. After artificial aging, the film samples were subjected to planned analyses and burial tests.

2.2.2. *Mechanical tests*

The tensile properties of the experimental films were measured according to the ISO 527-1:2019 and ISO 527-3: 2018 [36,37] testing methods. Mechanical tests were performed with a dynamometer Instron series 5900 at 23°C±1 and 50% RH at AUA for all samples.

2.2.3. *Chemical structure analysis*

Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) spectroscopic analysis was performed at AUA for BIOEL through Ingenio S by Bruker Optics, to identify the chemical composition and monitor the chemical structure changes in the film's external layers (4000-400 cm^{-1}; averages of 32 scans at resolution: 4 cm^{-1}). A single reflection diamond accessory, with refractive index 2.43, was employed. The penetration depth of the IR beam entering the diamond crystal, at 45°, is 1.66 μm for a wavelength of 1000 cm^{-1} [38].

Gel Permeation Chromatography (GPC) was carried out for BIOIT using a Malvern-Viscotek GPC max chromatographic system, equipped with a TDA 305 Tetra Detector system, pre-column and two columns Phenogel Phenomenex with an exclusion limit of 10^6 and 10^3 Da. A universal calibration curve was obtained by using 12 polystyrene standards with narrow polydispersity and a MW range of 0.5–3000 kDa. An isocratic elution of chloroform at a flow rate of 1 mL min^{-1} was applied.

2.2.4. *Pyrolysis GC/MS*

The Pyrolysis-Gas Chromatography-Mass Spectrometry (Py/GC-MS) analysis was used to identify the composition of the polymeric material. The identification of products evolving in the gas phase at a specific temperature and under an inert atmosphere, whose structure was determined by mass spectrometry in line with the GC apparatus, could be revealed with high sensitivity. MS analyses were performed at CNR-IPCB (Catania Unit, Italy). For the analysis, 0.1 mg sample was used. Pyrolysis was conducted in a multi-shot Pyrolyzer (EGA/PY-3030D, Frontier Labs), integrated with a GC-2020 system (Shimadzu Corporation) coupled with a triple quadrupole mass spectrometer (MS) detector utilizing electronic ionization at 70 eV (Mass Detector TQ8040, Shimadzu Corporation). The GC system featured an Ultra Alloyâ Metal Capillary Column (Frontiers Labs), with a stationary phase of 5% diphenyl-methyl polysiloxane, inner diameter of 250 μm, film thickness of 0.25 μm and length of 30 m. The interface temperature for Py-GC and GC-MC were maintained at 300°C and 250°C, respectively. To achieve a simplified chromatogram, a two-step temperature program was applied: 300°C and 400°C for sample analysis. The GC oven was initially set at 50°C for 1 minute, then ramped to 100°C at a rate of 30°C/min, held for 5 minutes, further increased to 300°C at 10°C/min, and finally reached thermal equilibrium at 300°C for 10 minutes. Helium was used as a carrier gas with a controlled flow rate of 1.78 mL/min, and a split ratio of 1:50 was applied. Blank runs were performed by pyrolyzing an empty crucible under identical conditions to eliminate background interference. Chemical structure is identified by matching the obtained spectra with reference spectra in the MS libraries (NIST11.Lib, NIST11s.Lib, WILEY8.LIB).

2.3. *Methodology for microplastics analysis*

Due to the current lack of reliable in situ methods for MP particle detection in soil, a series of soil processing steps for MP extraction from soil prior to analysis is required for reliable results. These typically involve the removal of the mineral fraction through density separation, followed by the elimination of natural organic matter via chemical or enzymatic digestion. The choice of soil processing methods must be made carefully to ensure the preservation of more labile synthetic polymers, which may be susceptible to degradation or loss during aggressive treatment procedures [39,40].

To optimise for potential loss of labile biodegradable MPs as a consequence of the extraction and purification procedure, soil samples from the experimental plots were processed using two different methods (*method I, method II*) that differ in the number of purification steps applied (Fig. 1).

In a first run, an aliquot of all soil samples was processed with density separation and enzymatic-oxidative purification in accordance with the soil protocol established at the University of Bayreuth (UBT) based on [40] and examined for MPs (*method I*). This protocol is specifically designed to avoid MP particle loss due to harsh treatment and allows for the detection of all standard plastic types including more labile polymers [39,40]. In a second run, another aliquot of soil samples taken under mulch films was processed with a minimum of soil processing steps required for the production of filters measurable with spectroscopy (*method II*). Here, mechanical stress was reduced to the lowest possible level to avoid potential particle disintegration of the labile biodegradable MPs by mechanical stress, e.g., mixing, shaking or filtering the sample. Both methods included gentle soil wet sieving through 500 μm mesh size. The fraction >500 μm was investigated visually under a stereomicroscope (Leica M50 with Olympus DP26 0.32x camera) and putative MPs (according to [41]) were sorted out with forceps. Each particle was photographed and its size measured. The chemical composition of the particles was then determined using ATR-FTIR (Bruker Alpha II, equipped with a diamond ATR crystal accessory, Bruker Optics GmbH & Co. KG, Ettlingen, Germany). The measurement parameters were set to an accumulation of 8 scans in the spectral range

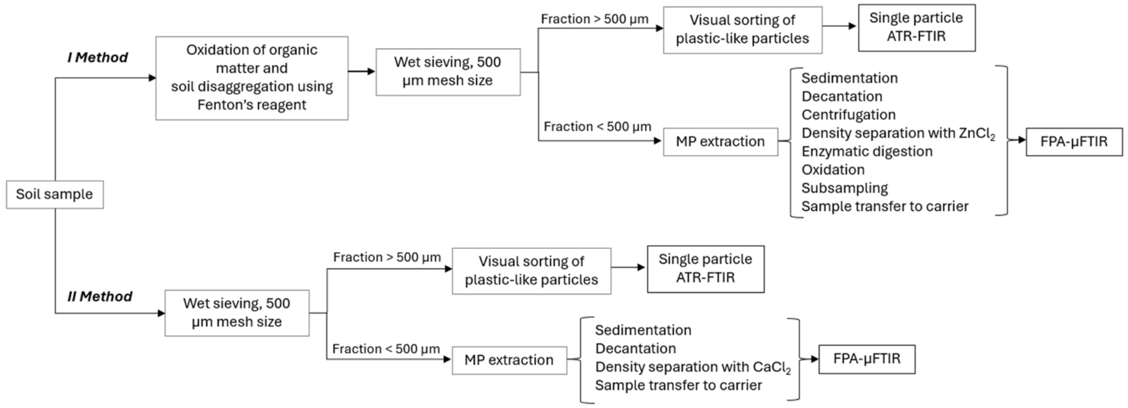


Fig. 1. Schematic representation of sample processing using two methods: (I) the full soil protocol was applied; (II) only the necessary steps were performed to isolate microplastics (MPs) from the soil.

from 4000 to 400 cm^{-1}, with a spectral resolution of 8 cm^{-1}. MP particles were identified by comparing sample spectra with an in-house generated polymer reference library [42]. Polymer type, size, shape and colour were recorded. The fraction below 500 µm was further processed through several consecutive steps, including sedimentation, decantation, and density separation, with enzymatic digestion and oxidation applied only in Method I. Finally, subsamples of remaining purified particles were filtered on Anodisc filters (pore size 0.2 µm; Ø = 25 mm; Anodisc, Whatman) with a glass funnel of 10 mm diameter and measured with focal plane array (FPA) based micro-Fourier Transform Infrared spectroscopy (µFTIR) with the IR microscope LUMOS II (Bruker Optics GmbH & Co. KG, Ettlingen, Germany). The microscope was equipped with an 8 x IR objective and liquid nitrogen cooled FPA detector with 32 × 32 pixels for chemical imaging resulting in a pixel size of 5.6 µm and a chemical image with about >3.5 million µFTIR spectra per filter. The entire particle-loaded area of the Anodisc filters was analysed in transmission mode on a CaF$_2$ transmission window (Ø = 25 mm, d = 2 mm). Samples were measured in the spectral range from 1250 to 3600 cm^{-1}, with a spectral resolution of 8 cm^{-1} and an accumulation of 1 scan per pixel. Background measurements were taken for each filter by imaging an area of the filter without sample. After converting the measurement data with the OPUS 7.5 software (Bruker Optics GmbH & Co. KG), automated particle identification and quantification were conducted using ImageLab (Epina GmbH, Retz, Austria) in combination with the commercial software tool Microplastics Finder, *Purency* (TU Wien – Innovation Incubation Center c/o Purency GmbH). This software tool is based on random forest decision classifiers as described in [43, 44], and automatically searches for IR spectra of the 22 most common synthetic polymers in the chemical image of the sample. The location, major and minor dimensions of the identified MP particles, color, shape, and polymer type were recorded. Each automatically identified MP particle was manually double-checked against reference spectra according to a four-eye principle by experienced personnel for quality assurance. A detailed description of sample preparation as well as quality assurance/quality control (QA/QC) procedures is provided in the SM Methods.

3. Experimental set-up of field tests

3.1. Protocol

The experimental protocol was designed to ensure consistency in sample handling, data collection and monitoring across the three field locations, with a focus on capturing baseline soil and environmental conditions.

The artificially aged BDMs were cut into standardized pieces measuring 10 cm x 21 cm. These pieces were enclosed in high-density polyethylene (PE-HD) mesh envelopes with a mesh size allowing soil contact while preventing loss of material during handling. The samples were buried at a depth of approximately 10 cm and retrieved following a randomized sampling order. Retrieval intervals varied, with almost monthly sampling in Greece and Italy, and adjusted schedules in Finland to account for frost periods. Upon retrieval, the samples were carefully cleaned without disrupting their content and photographed for disintegration image analysis.

Climatic and soil conditions were recorded systematically by sensors and data loggers during the trials to facilitate the analysis of the results. Meteorological data, including air temperature (°C) and precipitation (mm), were monitored. Soil conditions were also assessed continuously using sensors installed at a depth of 10 cm to record soil temperature (°C) and water content (WC, mass of water per unit mass of dry soil). The WC is expressed as a percentage of water holding capacity (% WHC).

3.2. Experimental fields

The full-scale experiments were conducted in Greece, Italy and Finland (SM, Fig. S1).

Greece: The field tests in Greece were carried out in the experimental field of AUA located in Spata, near Athens. The PE-HD envelopes, containing the biodegradable mulching film samples, were buried in the pit according to a randomized retrieval pattern. Meteorological data were recorded through the Spata Meteorological Station, located next to the study area. Soil temperature and WC were continuously monitored using the TEROS 11 sensor (METER Group, USA). Data was recorded at 30-second intervals and subsequently averaged over 5-minute periods.

To maintain optimal soil moisture, an advanced irrigation system equipped with a Raspberry PLC 42 controller (Industrial Shields, Spain) was deployed. The system utilizes 42 channels, comprising 26 input channels (12 analog and 14 digital) for real-time measurements and 16 output channels (10 digital and 6 digital/analog) for controlling operations, such as irrigation valves. The irrigation operated on a standard automated schedule, with a 15-minute watering cycles every 12 hours or whenever soil moisture fell below 60%. The functionality of the system was further enhanced by integrating weather data, such as rainfall, ensuring precise and adaptive water management to meet environmental and soil needs.

Italy: The field test was carried out at the experimental farm of UNIBA, in Valenzano (41° 01′ N, 16° 54′ E, and 124 m a.s.l., Bari, Southern Italy). Digital images of each specimen were taken before the burial.

The burial operations took place on 7 June 2022, burying the specimens 10 cm deep. Each specimen was embedded in a natural white PE-HD net (mesh of 2.7 mm x 7 mm; thread diameter of 0.29 mm) and labelled to be identified. The last sampling was after 870 days on 24

October 2024.

The sensors used to measure climate and soil parameters were: HygroClip-S3 sensors (Rotronic, Zurich, Switzerland) for air temperature and relative humidity; a pyranometer (model 8–48, Eppley Laboratory, Newport, RI, USA) for solar radiation on the horizontal plane; RT-1 sensors (METER Group, Inc., Pullman, WA, USA) for soil temperature; ECH2O 10HS sensors (METER Group, Inc., Pullman, WA, USA) for soil water content. Rain was monitored over time (ARIF, 2024). Soil temperature and water content sensors were placed at three different field points, 10 cm deep. The recording of the measurements started in June 2022 for the external environmental parameters and in July 2022 for the soil parameters. Measures were taken every 60 s, averaged every 15 min, and stored in the data loggers.

An automatic irrigation system was set up for regular cycles during summertime: watering was planned for 90 minutes, at 2.00 am once a week. About 30 liters of water per square meter were provided.

Finland: The field experiment in Finland was carried out at the research farm of Natural Resources Institute Finland (Luke) located in Jokioinen (60° 52′ 05.5" N, 23° 26′ 34.8" E).

The experiment started on 28 June 2022. The biodegradable mulching film samples enclosed in PE-HD mesh envelopes were buried to a depth of 10 cm. TMS-4 Standard dataloggers [45] were used to measure soil moisture using basic mode, which collected data at 15-minute intervals from a depth of 0-15 cm. ELOG9004 dataloggers were used to store soil temperature data 12 times a day from a depth of 10 cm. Meteorological data were recorded through Jokioinen Observatory. Following the prevailing agricultural management in Finland, no irrigation was applied as natural rainfall was sufficient to maintain soil moisture.

3.3. Soil characteristics

Soil analysis was conducted before the start of the experiments to establish baseline characteristics. The soil samples from all experiments were analysed in the same laboratory (Soil Science Laboratory of AUA) using the same methods to ensure consistency and results harmonization. The analysis included parameters such as soil texture and WHC, near saturated conditions, at pressures of 0 atm (pF0) [46], along with soil pH, Calcium Carbonate content (CaCO$_3$, %), total Nitrogen (N, expressed as a percentage of dry weight, Bucchi apparatus), Potassium (K, ppm), Phosphorus (P, ppm), Sodium (Na, ppm), total organic matter (% C of dry weight, Walkley-Black), Organic Carbon (%, Walkley-Black), electrical conductance (measured in μS/cm), and soil resistivity (Ω•m).

The soil characteristics of the three fields are presented in Table 2. The clay contents of the soil were similar (27-30%), whereas sand and silt contents varied more (Table 2). The pH values (1:1, v:v) indicate that the soils in Italy and Greece are slightly alkaline, while the soil in Finland is strongly to moderately acidic. Mineral arable soils in Finland tend to be acidic due to the parent material and climate conditions. To estimate the representativeness of the test field within the arable field soils in Finland, the soil pH was also analysed with the method used in agronomic soil testing and in national soil monitoring [47]. When measured from the soil:water suspension (1: 2.5, v/v) the pH was 5.9 and 6.1 in the 0-10 cm and 10-20 cm soil layers, respectively, thus falling within the range of pH reported for the mineral agricultural soils in the national soil monitoring of Finland [48]. Of the other measured soil properties, major differences are found in the lack of CaCO$_3$ and the high concentration of Phosphorus (Olsen-P) for the soil in Finland.

3.4. Experimental setup and sampling

The schedule of the (bio)degradation experiments in the three fields was organized based on weather conditions /season (Table 3). In Greece two series of experiments were carried out to confirm the results in different seasons and collect additional data.

Table 2
Soil characteristics & parameters of experimental fields.

Location	Spata (Athens), Greece	Valenzano (Bari), Italy	Jokioinen, Finland
Soil texture	Clay Loam	Sandy Clay Loam	Clay loam
Particle-size distribution [%] [49]	Clay: 27 Silt: 33 Sand: 40	Clay: 28 Silt: 18 Sand: 54	Clay: 30 Silt: 44 Sand: 26
CaCO$_3$ (%) wt/dry wt [50]	21.70	14.35	-
pH (H$_2$O, 1:1) [51]	7.78	7.43	5.4-5.9[1]
Total N (%) wt/ dry wt (Kjeldahl) [52]	0.168	0.2	0.192
Exchangeable K (ppm) [53]	648	806	402
P (ppm) (Olsen extraction) [54]	23.3	25.1	79.2[2]
Na (ppm) [53]	118	134	64
Total organic matter % wt/ dry wt (Walkley-Black) [55]	2.34	5.00	3.43
Organic C % wt/dry wt (Walkley-Black) [55]	1.05	2.00	1.72
Conductivity (μS/cm) [56]	1700	3700	1060
Soil resistivity (Ω•m) (Wenner method) [57]	5.8	2.7	9.4
Water Holding Capacity (%) [46]	43.0±2.4	54.5±3.2	48.1±4.7

[1] pH value of 5.9 measured by Luke

[2] Accuracy of the Method OLSEN is limited in acidic soils; more effective in extracting phosphorus in acidic conditions are the Bray-1 or Mehlich-3 tests (not available in the soil laboratory of AUA) [58].

Table 3
Full-scale field experiments plan for (bio)degradation of the biodegradable mulching films in soil.

Country	Film Code	Start date	Last sampling date	Retrieval	# of buried samples
Greece	BIOEL	10.04.22	10.09.2022	Monthly	36
	BIOEL	24.10.23	27.05.2024	Monthly	36
Italy	BIOIT	07.06.22	24.10.2024	During the first year, one retrieval was carried out every 40-50 days; afterwards one retrieval at the end of each summer	37
Finland	BIOEL	28.06.22	01.11.2024	Monthly excluding the months that the soil is frozen in Finland	36

In each field, during the designated time intervals, three mulching samples were retrieved in each sampling following the randomized collection plan. Then, the sample surface covered by soil was carefully cleaned with a soft brush (SM, Fig. S2a). Visible remains of fragmented samples could be found in soil aggregates collected from the AUA field during cleaning of samples that had started to degrade (SM, Fig. S2b). The retrieved samples were placed in a transparent plastic frame and photos were taken to evaluate their degree of disintegration with image analysis.

At the end of the experimental test, soil samples were collected to evaluate the presence of MPs inside the soil due to the burial of the materials. Soil samples were taken from the field at the layer 0-10 cm under artificially aged biodegradable mulch samples, randomly chosen. Each soil sample was placed inside a sanitised glass jar of 290 mL. Three samples of control soil were also collected, each taken from a different point of the experimental area far from the testing field. Soil was collected after 870 days of burial in Italy, after 216 days in Greece and after 862 days in Finland.

At the experimental fields, attention was paid to the clothes and gloves worn, which were made of cotton. Only the boots were made of plastic. Field equipment was washed with water and hexane/acetone to remove plastic residues or additive contamination.

4. Results

4.1. Artificial ageing induced photodegradation of biodegradable films

The evolution of the elongation at break (ε_{br}) of the BIOEL samples used in the two different periods of field experiments in Greece and in the experiment in Finland, as a function of their exposure time to artificial ageing at AUA, is shown in Fig. 2a. A fast degradation was observed, as the ε_{br} values of both series reached 50% of the initial ε_{br} values within 8 days. Similarly, BIOIT samples reached 80% of the initial value after 15 days of exposure to a Q-UV Panel equipment at CNR-IPCB. The characterization of the molecular weight of BIOIT film subjected to UV-radiation exposure highlighted the decreasing values of weight-average molecular weight (M_w) as measured by GPC. The evolution of M_w demonstrated the impact occurring during 15 days of the photo-degradation treatment on polymer M_w, which dropped by about 50%, reflected in an analogous way in the drop of the ε_{br} values (Fig. 2b).

4.2. Rainfall and soil water content

Random patterns in rainfall were observed during the three years with average rainfall being highest in Finland followed by Italy and Greece (Fig. 3a). During the year 2022, the summer in Finland was rather dry, in contrast with the summers of 2023 and 2024.

The soil WC, expressed as a percentage of the WHC (%WHC) of the corresponding soils, is presented in Fig. 3b. The soil WC of the field in Italy is quite similar to that of the field in Greece in terms of average values but varied between some seasons. The soil WC of the field in Finland is higher than that of the soil in the experimental field of Greece, except for the dry summer period of 2022 and June 2023.

4.3. Air temperature and soil temperature

The monthly mean values of air temperature, recorded in the locations of the experimental fields of Italy and Greece (Fig. 4a) were quite similar (max around 30°C in July and minimum around 10°C in January), as expected. The air temperature in the experimental field of Finland was much lower than that of Italy and Greece (max around 15°C in July and minimum around -10°C in December). The corresponding monthly mean soil temperatures recorded in the three locations are shown in Fig. 4b. The pattern of the soil temperatures follows that of the air temperatures for Italy and Greece. The maximum soil temperature in the field of Finland reached 20°C in July 2022, while it was below 0°C for a period of 4 months during winter, and below 5°C for 5 or 6 months.

4.4. Disintegration of mulching films in the soil of the field experiments

The disintegration, considered as an indication of biodegradation of the biodegradable mulching films in soil (Fig. 5), revealed significant differences in the degree and rate of disintegration between the three experimental locations (Fig. 6).

The artificially aged film BIOEL buried under natural soil conditions in the AUA experimental field completed the disintegration with a period of 5-7 months, depending on the season of exposure. During the samples' retrieval in the experimental field of Athens, after the 3rd month of exposure, a black thin layer (traces of carbon black powder) was visible just under the envelope's covering net (SM, Fig. S3).

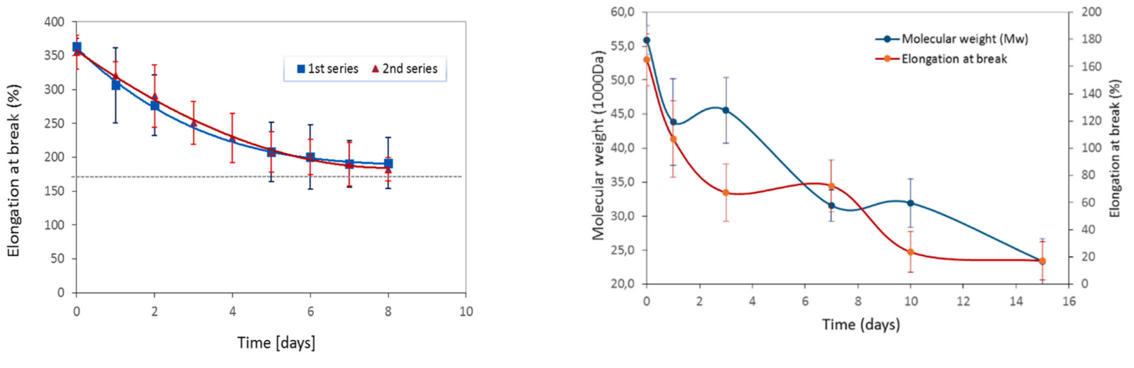
The same artificially aged BIOEL film, exposed under the field conditions of Finland, did not show complete disintegration over a period of 29 months, reaching an average degree of disintegration of 32%. The degree of disintegration of the samples exposed in the experimental field of Finland also shows high variability (SD) (Fig. 5 and Fig. 6).

The artificially aged BIOIT film buried under natural soil conditions in the experimental field of UNIBA showed a much slower rate of disintegration as compared to the case of disintegration due to biodegradation in Athens, achieving a degree of disintegration of 84.4% in 29 months.

4.5. Qualitative and quantitative analysis for MPs in soil

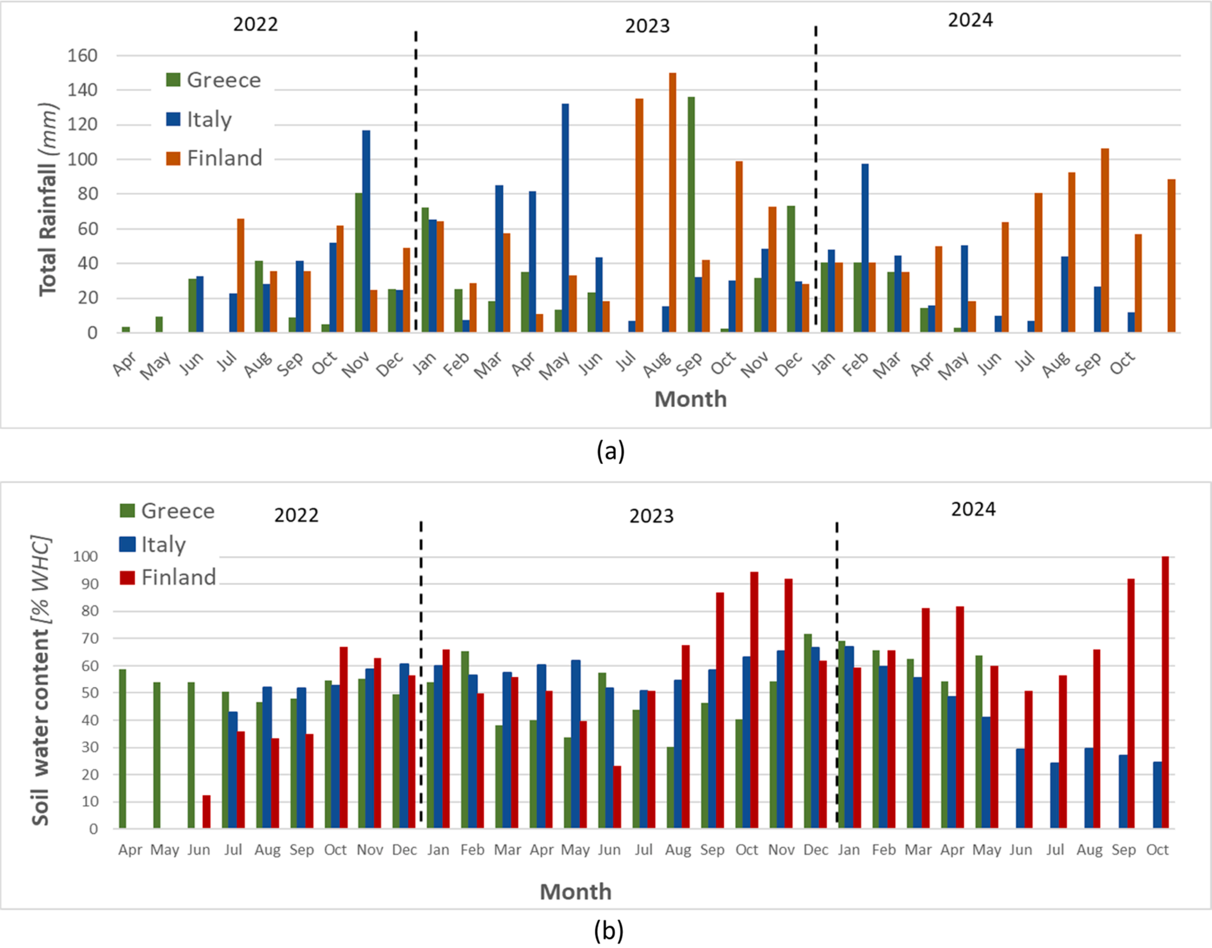
MPs from various types of polymers were detected in all samples analysed, with concentrations ranging from 700 to 24,200 MPs kg^{-1} dry weight (dw) (Table 4). Concentration of MPs originating from the fragmentation of applied soil biodegradable films BIOEL and BIOIT in the experimental fields varied between 0 and 7,022 MPs kg^{-1} (dw), corresponding to 0–70% of the total MP load. In Finland and Greece, where the BIOEL film was applied, MPs from biodegradable film accounted for less than 10% of the total identified MP load. In contrast, in Italy, where the BIOIT film was used, MPs from the film comprised a significantly higher proportion, reaching up to 70% of all detected particles.

Comparing the two microplastic extraction and purification methods (*method I and II*) that differ in the number of soil treatment steps, it was observed that the number of biodegradable film fragments was low when the full soil processing protocol was applied (Table 4). In the samples from Italy, nine particles could be attributed to biodegradable film fragments, while four particles were identified in the samples from Greece and none in the Finnish samples. In contrast, when oxidation and enzymatic digestion were left out to purify MPs from soil organic matrix,



(a) (b)

Fig. 2. (a) Evolution of the elongation at break (ε_{br}) for BIOEL with respect to time of exposure to artificial ageing (AUA). (b) Change of weight-average molecular weight (M_w) and ε_{br} of BIOIT with time of exposure to artificial ageing (IPCB-CNR).



(a)

(b)

Fig. 3. Total rainfall (a) and soil WC (b) in the three experimental field locations in Greece, Italy and Finland.

significantly more biodegradable film fragments were detected in the samples from Italy (in total 2508 particles). However, this approach did not result in a significantly increased number of detectable mulch film fragments in samples from Greece and Finland, where the BIOEL mulch film was applied. Only six mulch film fragments were detected in soil samples from Finland and five in samples from Greece, respectively. The majority of mulch film fragments were detected in the larger size fraction >500 μm, which was not subjected to density separation.

MPs from 19 different polymer types were detected across all samples analysed from the three countries involved in the study: Finland (13 polymer types), Greece (17), and Italy (15) (SM, Table S1). When excluding particles originating from biodegradable films, background MP pollution in each country was predominantly characterized by the presence of polypropylene (PP) fragments, which emerged as the most frequently occurring polymer type (SM, Fig. S4). PE, polyethylene terephthalate (PET), and polystyrene (PS) were also consistently found among the most abundant polymers detected, comprising the next highest proportions of MP particles across the samples. Fibers accounted for approximately 2.2% of all particles, while the remaining 97.8% were classified as fragments (SM, Table S1).

The FTIR spectra of black mulch film fragments isolated from soil samples from Italy (samples IT-BIOIT-1-3), Finland (samples FI-BIOEL-2-3) and Greece (EL-BIOEL-2), compared to reference spectra of aged BIOIT and BIOEL mulch films, are presented in Fig. 7a,b. Images of biodegradable film fragments are displayed in Fig. 7c. Fragments can be characterised as cracked, black-greyish, brittle pieces that tend to disintegrate even when carefully handled with tweezers.

The particle size distribution of biodegradable film fragments compared to all other MPs is shown in Fig. 8. Background pollution was represented predominantly by smaller particles (<500 μm), while biodegradable film fragments in Finland and especially in Italy were represented by larger particles (>500 μm). A small percentage (<1%) of macro particles of biodegradable films (>5 mm) was detected only for BIOIT. All detected BIOEL particles in Greece and Finland belong to the micro particle class (<5mm).

5. Discussion

The identification of environmental parameters acting as inhibition factors and possible soil pollution by MPs related to disintegration due to biodegradation of certified soil biodegradable mulching films in Mediterranean and Northern climates is discussed in this section, based on the analysis of the results presented in the previous section.

5.1. The effect of the soil water content

It is noted that soil WC ≤ 20 (% WHC) has been associated with no disintegration, or very slow disintegration of cellulose [25]. No inhibition of biodegradation and measured disintegration was expected in the three experimental fields due to low soil WC, except for June 2022 and marginally June 2023, for Finland (WC ≤ 20 % WHC).

On the other hand, the highest soil WC was measured in Finland and the WC increase in the autumn coincided with the decreasing temperatures. Excess wetness contributing to water saturated conditions can reduce the biological activity in soil [59,60]. The high moisture content and possible formation of anoxic conditions in part of the pore space in the soil could partly explain the high variability of disintegration within the experimental field in Finland.

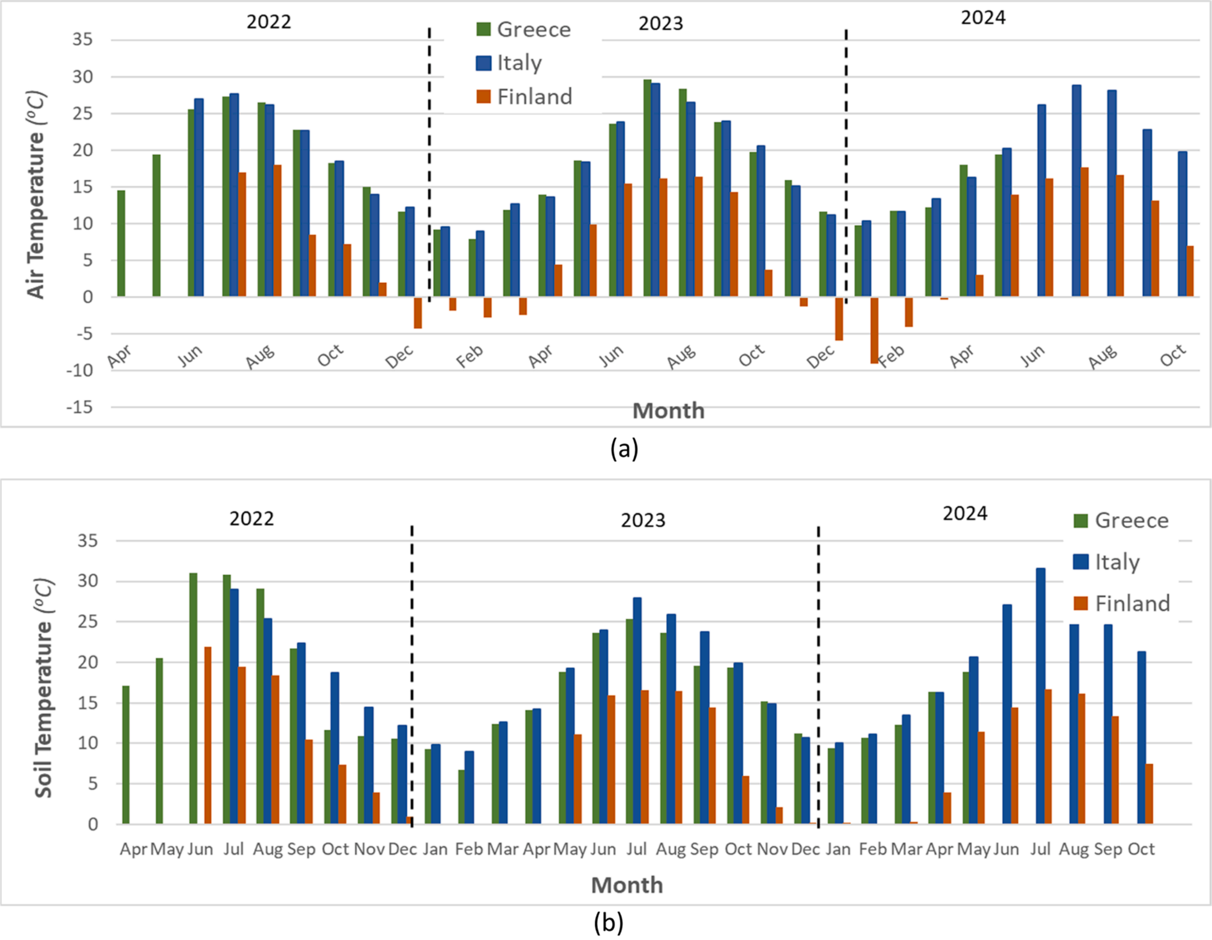


Fig. 4. Monthly mean air temperature (a) and soil temperature (b) in the three experimental field locations in Greece, Italy and Finland.

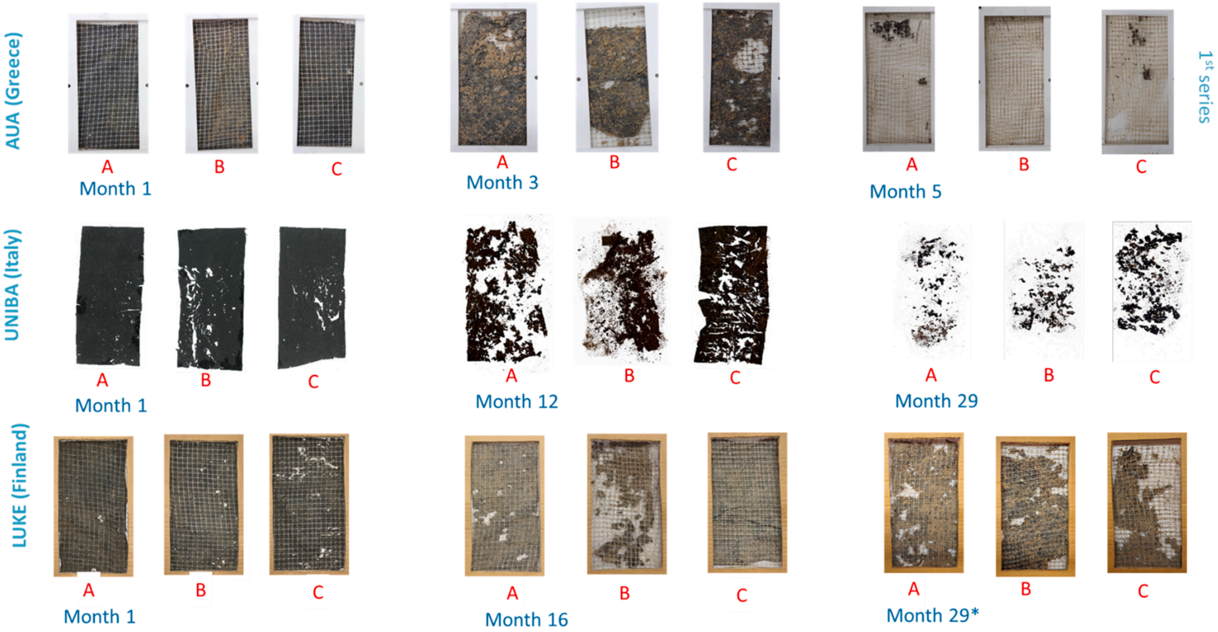


Fig. 5. Evolution of disintegration in soil with time of biodegradable-mulching films during the field experiments in Greece, Italy and Finland.

5.2. The effect of the soil temperature

In general, biological activity in soil decreases as the soil temperature approaches zero [59,61], and no disintegration was observed for cellulose for soil temperatures below 5°C [25]. Thus, the soil temperatures below 5°C for 5-6 months (from which below 0°C for 3-4 months) in

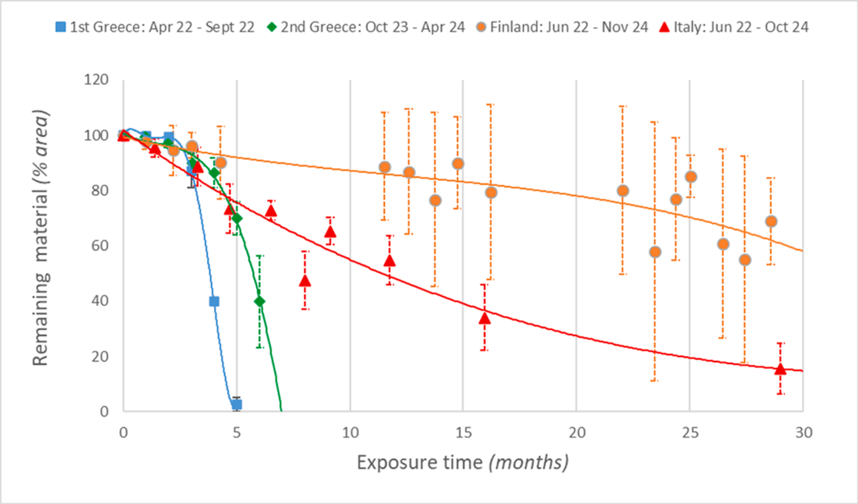


Fig. 6. Degree and rate of disintegration of artificially aged biodegradable mulching films in Greece, Italy and Finland.

Finland can be considered as an inhibiting factor for disintegration due to biodegradation. Similarly, biological activity increases with temperature, and a 10°C rise has been suggested to result in a twofold increase in organic matter mineralisation [62]. Soil temperatures in Finland were generally about 10°C lower than in Italy or Greece, indicating significantly lower mineralisation rates in Finland during most of the degradation experiment.

5.3. *The effect of the soil pH*

The soil pH measured in Finland was lower than in other test locations and indicated moderate (pH 5.9) or strong (pH 5.4) acidity of the soil (Table 2) [63,64]. The degradation test in the Boreal climate was done with the same material as in Greece. The conditions in Finland differ from those in Greece in relation to climate and soil pH. Within northern Europe, cool climate and low soil pH are tightly linked. In Scandinavia, two natural factors favor the development of acid soil: the presence of predominantly crystalline acid rocks combined with the absence of carbonate rocks and the climate favoring organic matter build-up and leaching of basic cations [65]. Soils with low pH levels are typically found in regions with a Nordic climate, characterized by cold winters and mild, humid summers [66]. Therefore, the results reflect the differing degradation patterns between South and North Europe in typical natural environment, considering climate and soil conditions, providing a comprehensive evaluation of BDMs performance in different geographical locations. A slow disintegration in Finland was indeed expected qualitatively (no quantitative prediction was possible however), mainly because of the prevailing lower temperatures (November to April <5 °C, partly below 0 °C). During the period May to October, the average temperature was 14 °*C* (max temperature 20 °C). The acidic soil, on the other hand, is considered as a major inhibition factor for the whole period of the experiment, as it is strongly related to reduced microbial activity, according to multiple sources of the literature.

It is well known that the variation of soil temperature is a critical driver of the biodegradation rate of plastics, with warmer conditions typically enhancing microbial activity and accelerating the degradation process, while colder temperatures significantly slow it down. Biodegradation rates in soil often exhibit an exponential relationship with temperature within the mesophilic range (15–28°C), which can be accurately described by the Arrhenius equation [67]. Seasonal shifts significantly affect degradation. Studies show that biodegradable mulch films (e.g., PLA/PHA blends) biodegrade more rapidly during summer months compared to winter, directly correlating with warmer soil temperatures [68]. In that study, it was found that the rate of PHBH biodegradation increased with increasing temperatures in three different soils, but that the rates and temperature dependence of the rates varied between soils. This suggests that in the case of the field experiment in Finland, the acid soil has also contributed to the extremely slow degradation rate.

Low soil pH, though characteristic of the boreal climate zone in EU [69] is known to affect soil biological activity. According to literature, reduced activity has been linked to both direct impacts on microbial biomass and indirect effects through decreased soil productivity, which lowers substrate input [70]. These soils show increased solubility of zinc (Zn), manganese (Mn), aluminium (Al) and/or iron (Fe), which can lead to rapid accumulation and toxicity. The dissolved aluminium Al^{+3} is a dominant problem in acidic soils, because it is toxic to plants, however, causing toxicity symptoms in general in pH values lower than 5 [71]. The oxidative stress induced by aluminium (Al^{3+}) also affects soil microbial activity negatively, including the decomposition of organic matter and mineralization [70,72,73]. The low availability of phosphorus and molybdenum (one of the essential micronutrients) to plants due to low pH also affects nitrogen fixation and nutrient deficiencies in calcium (Ca) and magnesium (Mg) [74,63,73,75]. In addition to reducing productivity, low pH may limit substrate availability in soil by decreasing the solubility of soil organic matter. A consistent positive relationship between soil pH and dissolved organic matter, a key substrate for soil biological processes was reported in Evans et al. [69]. The reference to the above validated literature results supports qualitatively the observed disintegration behaviour of the biodegradable mulching film BIOEL in Finland. Analytical investigation of these parameters was beyond the scope of the present work.

5.4. *Comparative analysis of the impact of soil and climatic factors on disintegration behaviour*

The disintegration behaviour of the artificially aged biodegradable mulching films buried in the soil of the experimental fields in Italy, Greece and Finland (Fig. 6) is comparatively analysed considering the effects of the main experimental parameters discussed in the previous sections, and summarized in Table 5.

It is apparent that the soil texture is not a crucial parameter in this comparison as all soils belong to the general class of clay loam, including sandy clay loam. On the opposite, the locations of the disintegration experiments differed according to soil pH, temperature and moisture. The soil in Finland was acidic differing from the alkaline soils of Italy and Greece. Low pH is likely contributing to the low biodegradation as it is associated with a risk of increased aluminium solubility, and nutrient

Table 4
MPs concentrations in soils from experimental fields in Finland, Greece and Italy.

Country	Sample treatment[1]	Sample name	Soil mass processed, g	Total MP count per kg soil (dw)	Total count of bio-film MPs per kg soil (dw)	Count of bio-film MPs (>500 µm) per kg soil (dw)	Count of bio-film MPs (<500 µm) per kg soil (dw)	Proportion of bio-film- MPs from total, %
Finland	I	FI-Control-1	20	1200	0	0	0	0
		FI-Control-2	20	700	0	0	0	0
		FI-Control-3	20	1200	0	0	0	0
		FI-BIOEL-1	20	2350	0	0	0	0
		FI-BIOEL-2	20	1200	0	0	0	0
		FI-BIOEL-3	20	3300	0	0	0	0
	II	FI-BIOEL-1	141	1191	0	0	0	0
		FI-BIOEL-2	149	875	13 (2)	13 (2)	0	2
		FI-BIOEL-3	141	1841	78 (4)	21 (3)	57 (1)	4
Greece	I	EL-Control-1	20	9800	400 (1)	0	400 (1)	4
		EL-Control-2	20	7900	0	0	0	0
		EL-Control-3	20	8000	0	0	0	0
		EL-BIOEL-1	20	12100	0	0	0	0
		EL-BIOEL-2	20	18200	200 (4)	200 (4)	0	1
		EL-BIOEL-3	20	14500	0	0	0	0
	II	EL-BIOEL-1	116	4321	0	0	0	0
		EL-BIOEL-2	78	6927	513 (5)	0	513 (5)	7
		EL-BIOEL-3	136	1367	0	0	0	0
Italy	I	IT-Control-1	20	12800	0	0	0	0
		IT-Control-2	20	17000	0	0	0	0
		IT-Control-3	20	24200	0	0	0	0
		IT-BIOIT-1	20	8700	800 (2)	0	800 (2)	9
		IT-BIOIT-2	20	4150	50 (1)	50 (1)	0	1
		IT-BIOIT-3	20	11650	300 (6)	300 (6)	0	3
	II	IT-BIOIT-1	172	4825	2413 (401)	2320 (399)	93 (2)	50
		IT-BIOIT-2	178	9980	7022 (1229)	6888 (1226)	135 (3)	70
		IT-BIOIT-3	156	10323	5718 (878)	5615 (876)	103 (2)	55

[1] I – soil samples processed according to full soil protocol, II – Soil samples processed according to a shortened soil protocol that only included sieving and density separation; in parenthesis – the number of physically and chemically detected particles

deficiencies reducing the microbial activity and therefore affecting negatively the biodegradation /disintegration process.

The soil WC (%WHC) varied with season, in the range of 30-72% for Greece, 24-67% for Italy and 13-100% for Finland. These soil WC variations were expected, considering also that the fields in Greece and Italy were irrigated during the summer period. The exception was June of 2022 and 2023 in Finland, when the low WC may have inhibited the biological activity. It is also interesting that in Finland, the soil WC in the fall reached high levels close to WHC, possibly resulting in anoxic conditions in localized parts of the field that inhibited biodegradation/ disintegration.

The soil temperature also varied with season, in the range of 7-31°C for Greece, 9-32°C for Italy and below 0°C up to 20°C for Finland. The variations of soil temperature in Greece and Italy as well as low temperatures in the field of Finland were expected and considering the significant impact of temperature on biological activity as discussed in 5.2, support the degradation results.

Considering the above analysis of the main parameters affecting the disintegration/ biodegradation of the mulching films in the three locations, in the case of Finland, the main inhibiting factor was the low winter temperatures for a period of 5-6 months and the overall lower soil temperature during the other months. The overarching inhibiting factor in the Finnish experimental field, however, contributing to the slow disintegration of the mulching film throughout the study period, was the low soil pH, as it is strongly related to reduced microbial activity, according to multiple sources of the literature.. The combination of the 2

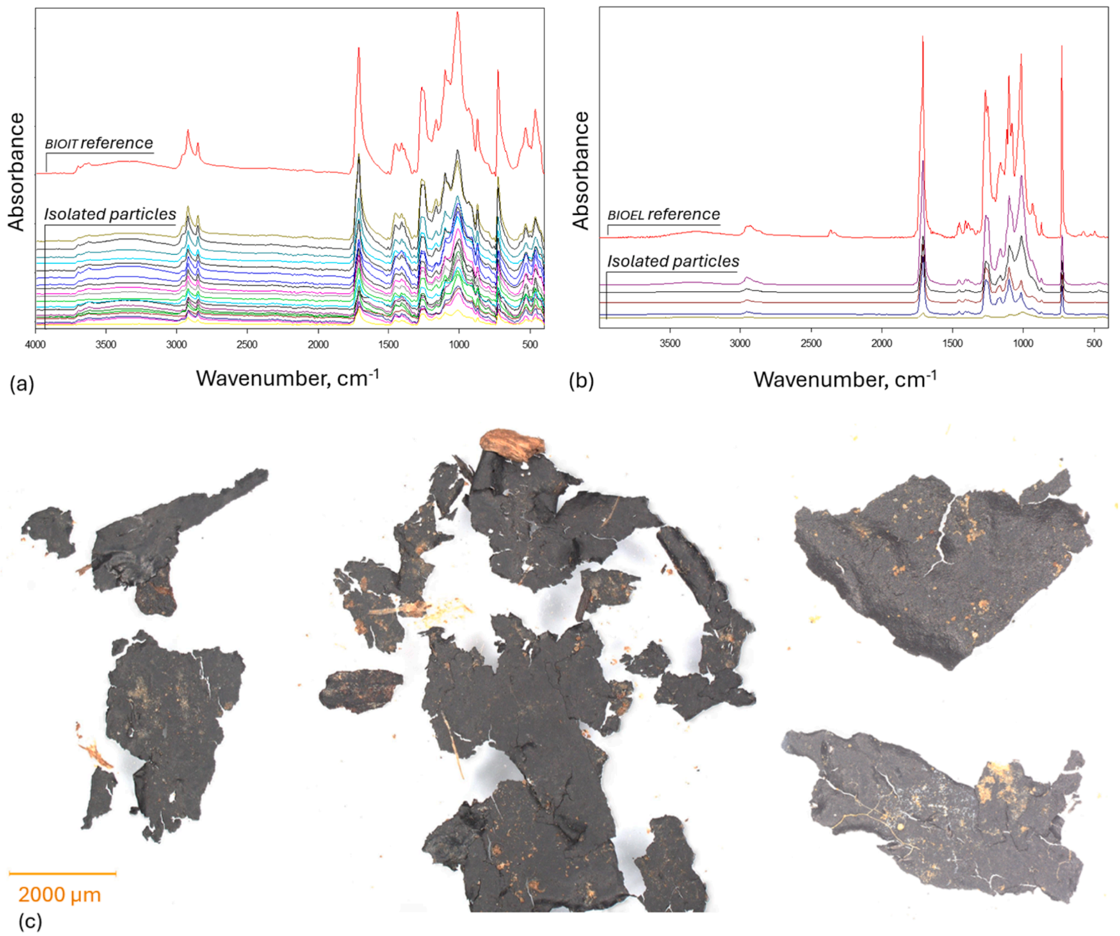


Fig. 7. FTIR spectra of isolated black particles from samples IT-BIOIT-1-3, FI-BIOEL-2-3 and EL-BIOEL-2 compared to the FTIR spectra of the reference films (red): a) BIOIT; b) BIOEL. c) Images of biodegradable film fragments isolated from the sample IT-BIOIT.

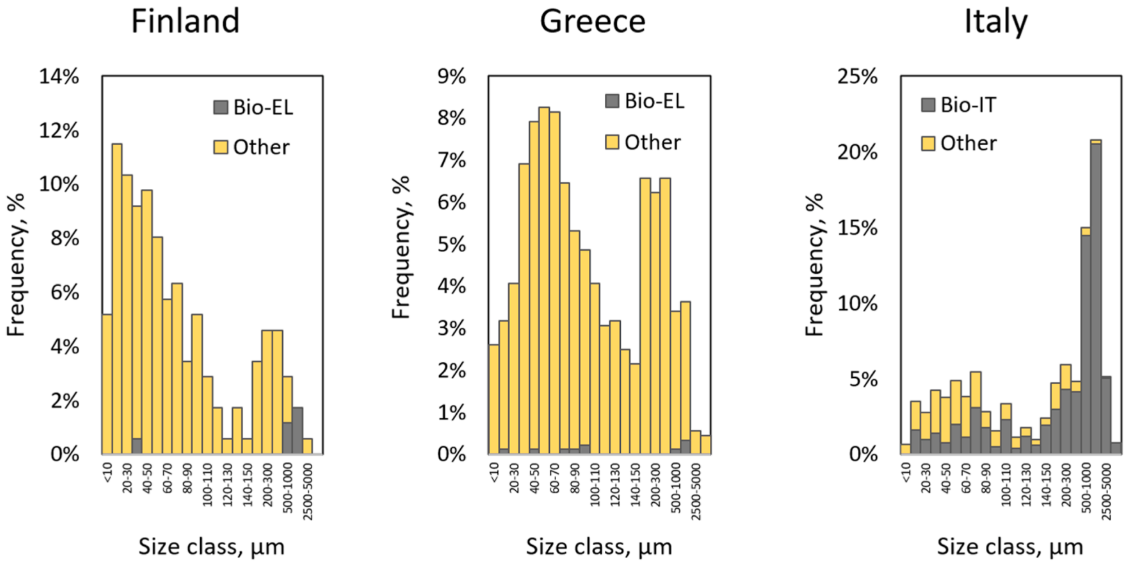


Fig. 8. Size distribution of biodegradable film fragments compared to all other MPs.

dominant inhibiting factors, identified in the case of the field experiment in Finland, along with the partial contribution of the dry (2022) and high WC (2023-24) periods, explains well the slow disintegration rate observed, achieving a degree of disintegration of only 32% after 29 months of burial exposure.

What is not explained, however, in terms of the analysis of the main parameters affecting the disintegration/ biodegradation of the mulching films, is the relatively slow degree of disintegration of the mulching film in the experimental field of Italy as compared to that recorded in Greece, under similar environmental conditions. As similar field experiments

Table 5
Main experimental parameters contributing to the disintegration behaviour of the biodegradable mulching film in the three locations.

Region	Soil texture / pH	Soil water content *(%WHC)*	Soil temperature *(°C)*	Remarks	Degree of Disintegration *(%)* *(months of exposure)*
Finland	Clay loam 5.38	12-100	≤ 0 – 20	Acidic soil contributing to reduced microbial activity Avg. WC: 61.3 *(%WHC)* Avg. soil temperature: 10.0 °*C,* (Avg from May to October: 14 °*C*; temperature from November to April <5 °*C*, partly below 0 °*C*) Dry soil (low WC) in summer 2022, combined with winter freezing soil temperature, several months below 5°C	32 *(29)*
Greece	Clay Loam 7.78	30-72	7-31	Avg. WC: 52.0 *(%WHC)* Avg. soil temperature: 17.3 *°C*	100 *(5-7)*
Italy	Sandy Clay Loam 8.09	24-67	9-32	Avg WC: 51.1 *(%WHC)* Avg. soil temperature: 19.3 °*C*	84.4 *(29)*

were conducted in the same experimental fields of Greece and Italy several years ago, it was considered important to briefly review those results against the present ones.

5.5. The disintegration rates compared against earlier experiments in the experimental fields of Greece and Italy

Greece: The two series of biodegradation tests of the artificially aged BIOEL film carried out in the experimental field of AUA at different seasons explain the small differences in the recorded time for full disintegration. The first test, carried out during the period April-September 2022, was ideal for biodegradation under high temperatures in an irrigated field, reaching full disintegration in 6 months (Fig. 6). The second series, October 2023 – May 2024, included the winter period with prevailing low temperatures and reached full disintegration in 7 months.

It is interesting to note that the disintegration behaviour of the 2 series of the artificially aged BIOEL film buried in the experimental field of AUA was found to agree very well with the results obtained in the same field with similar starch/PBAT commercial mulching films (but different grades), with watermelon cultivation in the framework of the BIOPLASTICS project, in 2001-2005 (Fig. S5).

Italy: The biodegradation tests of the artificially aged BIOIT film carried out in the experimental field of UNIBA were extended for more than two years without full disintegration and included 3 summer periods with rather favourable conditions for biodegradation in soil. By comparing this behaviour against the corresponding disintegration behaviour of BIOEL in AUA under similar climatic and soil conditions, the slow disintegration behaviour of BIOIT was characterized as "unexpected".

UNIBA carried out field experiments with two starch-based biodegradable mulching films (50 μm and 25 μm thick) at the Agricultural Experimental Station in Policoro (Matera, Italy) under the same BIOPLASTICS project (Reference S3). Regarding the film composition the only information provided was that the starch-based raw material was a starch/PBAT blend similar to the one tested in Greece by AUA. At the end of the cultivation period of 9 months, the biodegradable films were broken into very small pieces and buried in the soil onsite together with the plant residues and afterwards, no activities were carried out on the soil. Soil samples were taken periodically from 1 m^2 of field surface at a depth of 0.20 m. The residues of biodegradable film not passing through a 1.8 cm mesh were collected and weighed. One year after the burial, less than 4% of the initial weight of the two biodegradable films was found in the soil. The scope of that study did not include analysis of the soil for the presence of MPs [76]. Those earlier results agree better with the earlier and the current results in Greece, and they are not in agreement with the current results of BIOIT, which showed a much longer disintegration period. As this low degradation of BIOIT in Italy was not supported by the earlier research results in the same field, a more detailed investigation of the composition of the biodegradable films used in Greece and in Italy was conducted.

5.6. The effect of the composition of the biodegradable mulching films

5.6.1. Analytical identification of composition

The advanced analytical protocol employed by CNR-IPCB through Py/GC-MS, described in the methodology section, allowed to determine the polymeric composition of pristine BIOEL and BIOIT samples, including polymeric nature additives.

The Py/GC/MS chromatograms of pristine BIOEL and BIOIT shown in Fig. 9, reveal a relevant difference in the formulation of the two biodegradable films. The chromatograms of BIOIT collected at 400°C and confirmed with the mass spectra of NIST library with a match of 97%, suggest that the peak at 10,8 min belongs to 3,6-dimethyl-1,4-dioxane-2,5-dione (DL-Lactide) derived from degradation of PLA. This peak was not revealed in the chromatogram of BIOEL samples.

The unexpected deviation in the composition of BIOIT was detected at the end of the field experiments in Bari, in an effort to understand the different behaviour of BIOIT from that of BIOEL films. The presence of PLA in the formulation of BIOIT film implies that this film was not exactly as described in Table 1 by the supplier, but a slightly different material.

The presence of PLA in the composition of the BIOIT mulching film explains perfectly well the slow rate of its disintegration as compared to the BIOEL mulching film, even though they were exposed to similar soil/environmental conditions. At the beginning of the present research work, both BIOIT and BIOEL were claimed by the supplying industries to be based on the same raw material certified as biodegradable in soil (TDS by manufacturers, Table 1). However, it was the composition analysis that revealed a differentiation in the composition of the BIOIT film, with the presence of PLA, finally supplied by the industry in Italy. This was due to a mistake (according to the manufacturer), that supplied the partner UNIBA with a different biodegradable material grade than the film claimed in Table 1. This mistake led to the unexpected research outcome of the field experiment in Italy. On the other hand, it also confirmed major inhibition effects of specific polymeric compositions, such as the BIOIT film, resulting in slow rates of disintegration. Lack of accurate information about the polymers used in a material's composition (without disclosing any details) is a major obstacle to the proper function and evaluation/certification of commercial mulching films.

5.6.2. MP analysis

From an analytical perspective, biodegradable plastics pose significant challenges, as they are designed to undergo degradation under natural environmental conditions. This inherent degradability can complicate their detection, identification, and quantification during MP analysis, particularly when using conventional purification methods that may further alter or degrade these materials. MP particle analytical detection was particularly challenging in the case of biodegradable mulch film fragments from both film types BIOIT and BIOEL, as the

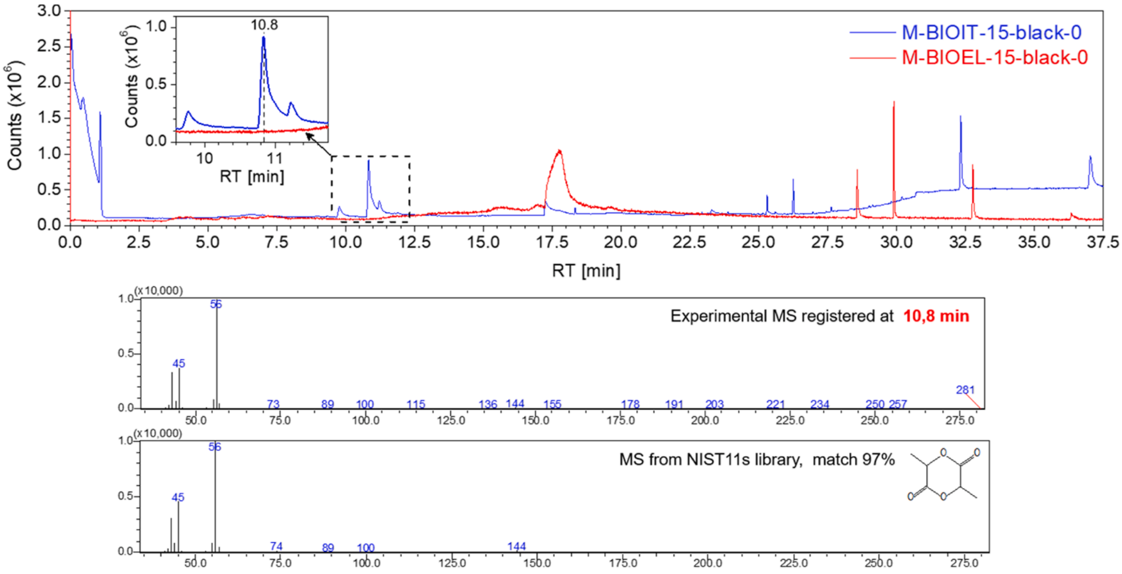


Fig. 9. Py-GC-MS of pristine BIOIT (blue line) and BIOEL (red line) collected at 400°C. MS spectrum of peak at 10.8 min and the match with the NIST library corresponding to the 3,6-Dimethyl-1,4-dioxane-2,5-dione (DL-Lactide) derived from degradation of PLA.

fragments exhibited high brittleness and a strong tendency to disintegrate even under minimal mechanical stress. This is particularly obvious when comparing results from Italy obtained from experiments using the full and the adjusted reduced soil processing protocols (*methods I and II*). Over two thousand BIOIT film MP fragments were isolated from soil samples following the sieving and removal of only the mineralogical soil fraction. In comparison, only a few MP fragments were recovered from the same soil samples subjected to a full treatment protocol. This suggests that minimal processing only may be necessary for preserving and isolating BIOIT particles from soil matrices. The majority of BIOIT film fragments were likely disintegrated or degraded during successive sample enzymatic purification steps and were subsequently lost during filtration. In contrast, soil samples from Finland and Greece where BIOEL films were tested and which were processed according to a shortened soil processing protocol allowing for maximum preservation and isolation of brittle fragments, still did not reveal a significantly higher number of detectable mulch film MP fragments. This suggests that the BIOEL mulch film fragments in both countries were probably present in soil in much smaller amounts prior to analysis in contrast to the experiment in Italy where BIOIT film was tested. This observation confirms the slow degree of disintegration of BIOIT due to its different composition, compared to BIOEL, resulting in apparent inhibition effects (Fig. 6).

Most of the biodegradable film MP fragments detected in the soils were larger than 500 μm. Even in the samples from Italy, which underwent only gentle sieving and density separation, only a few smaller than 500 μm particles were detected, despite the high abundance of fragments larger than 500 μm. Given that only chemically neutral solvents and solutions were used in a shortened protocol, the most probable cause of particle loss could be the mechanical disintegration of fragile fragments during gentle mixing with the density solution. Unfortunately, with the current state-of-the-art methods, reliably detecting such brittle particles smaller than 500 μm remains challenging, especially since density separation is a critical step in the extraction and identification of MPs from soil.

Although exclusion of such vital soil processing steps as soil aggregate disintegration and organic matter digestion prior MP analysis favoured the detection of many BIOIT film fragments, this method hinders efficient and quantitative particle extraction and detection. For example, particles incorporated within soil aggregates may not be effectively isolated and are likely lost during the density separation process, as they sediment together with the mineral fraction (e.g. SM Fig. S2b). Additionally, the presence of the soil organic matrix can interfere with spectroscopic detection by physically obscuring or entrapping MPs, thereby hindering their identification. This effect is particularly evident in the reduced MP background contamination observed when comparing results obtained from both analytical methods. Thus, the results of the MPs analysis in soil samples processed according to the full soil treatment protocol probably underestimate the amount of biodegradable mulch film fragments, but reliably represent background MP pollution with the majority of polymer types.

6. Conclusions

Agricultural plastics, extensively used worldwide, offer multiple benefits to agriculture. However, their use is also associated with negative impacts, with the mismanagement of agricultural plastic waste being a dominant one. In addition, several knowledge gaps need to be investigated in depth. Included among them are possible inhibition factors on the biodegradability of bio-based biodegradable in soil mulching films related to different climate zones, environmental conditions, and composition of the materials, and the relationship of these factors to possible soil pollution by MPs of the biodegraded mulch films. The interrelationship of the inhibition factors due to MP-induced soil pollution is largely unknown due to limited and focused comparative full-scale studies.

The comparative disintegration (as an indication of biodegradation) of artificially aged samples of commercial starch/PBAT based mulching films, certified as biodegradable in soil, investigated under field conditions in Southern and Northern Europe, revealed the specific role of crucial climatic factors, soil characteristics, and materials composition, responsible for significant differentiation of disintegration rates. Of the measured environmental parameters affecting the disintegration of the mulching films, the main differences between the three locations were soil temperature interrelated with soil chemical properties (mainly pH) and moisture.

The two dominant inhibition factors for disintegration of mulching films in soil, identified under the Northern European climate and soil conditions of Finland include: a) Low soil temperatures, less than 5°C, recorded for 5-6 months, from which 3-4 months below 0°C, and also 10 degrees lower temperatures in summer than in Southern Europe; b) Strongly to moderately acidic soil, impeding the microbial activity according to literature. These factors, combined with an unusually dry summer period in 2022 and high soil moisture levels in autumn and spring of 2023, and 2024, explain well the very slow disintegration rate observed, achieving a degree of disintegration of 32% after 29 months of burial exposure. Similar behaviour should be expected for other geographical locations with low temperatures prevailing during the wintertime, in combination with acidic soil.

The mild soil temperatures prevailing in Southern Europe, along with normal soil water content of the irrigated field led to full disintegration of the same film tested in the field experiment of Finland, much faster in Greece, in 5-7 months. The disintegration rate of a similar film in Italy, under analogous climatic conditions to those of Greece, was significantly lower (84% in 29 months). This unexpected behaviour resulted from the inhibition effects caused by an identified deviation of the material composition of the specific film from the composition of the film used in Finland and Greece. This was the case with the slow disintegration rate of the film tested in Italy with a degree of disintegration of 84.4% in 29 months.

The analysis of MPs in the three experimental fields at the end of the experiments revealed a low number of MPs originating from the biodegradable film exposed in Greece and Finland, as compared to a large number of MPs originating from the different mulching films exposed in Italy, confirming the inhibition effect of the particular composition that resulted in higher MP soil contamination.

The results obtained show the need for the development of materials, designed to be biodegradable in soil under different climate zones and soil environments, such as innovative biodegradable mulching films of special composition that could biodegrade under low temperatures and acidic soils, and certified accordingly. The development of new grades of biodegradable materials, designed to be functional and biodegradable under different climate zones and soil environments, without generating soil pollution by MPs represents a real challenge that needs to be addressed urgently.

CRediT authorship contribution statement

Demetres Briassoulis: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Christina Pyromali:** Writing – review & editing, Validation, Methodology, Investigation, Formal analysis, Data curation. **Fabiana Convertino:** Writing – review & editing, Validation, Methodology, Investigation, Data curation. **Evelia Schettini:** Writing – review & editing, Validation, Supervision, Methodology, Investigation, Formal analysis, Data curation. **Sabrina Carola Carroccio:** Writing – review & editing, Supervision, Methodology, Investigation, Formal analysis. **Pierfrancesco Cerruti:** Writing – review & editing, Supervision, Methodology, Investigation, Formal analysis. **Sarmite Kernchen:** Writing – review & editing, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Sandro Dattilo:** Writing – review & editing, Investigation, Formal analysis, Data curation. **Johanna Nikama:** Writing – review & editing, Methodology, Investigation, Data curation. **Helena Soinne:** Writing – review & editing, Validation, Methodology, Data curation. **Giuliano Vox:** Writing – review & editing, Investigation, Data curation. **Salla Selonen:** Writing – review & editing, Methodology. **Martin G.J. Löder:** Writing – review & editing, Methodology, Investigation, Data curation. **Christian Laforsch:** Writing – review & editing, Methodology. **Antonis Mistriotis:** Writing – review & editing, Validation, Methodology, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.polymdegradstab.2026.112163.

Data availability

No data was used for the research described in the article.

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