

The environmental behavior and fate of micro ^{14}C -polylactide in soils

Sara Adeleh^{a,b,d,*}, Tabea Becker^{b,d}, Sonja Herres-Pawlis^{b,d}, Roland Bol^{a,d},
Birte Drewes^c, Thomas Pütz^{a,d}

^a Institute of Bio and Geosciences (IBG-3: Agrosphere), Forschungszentrum Jülich GmbH, 52425, Jülich, Germany

^b Institute of Inorganic Chemistry, RWTH Aachen University, 52074, Aachen, Germany

^c Institute of Neuroscience and Medicine (INM-5: Nuclear Chemistry) Forschungszentrum Jülich GmbH, 52425, Jülich, Germany

^d Bioeconomy Science Center (BioSC), Forschungszentrum Jülich GmbH, 52425, Jülich, Germany

ARTICLE INFO

Keywords:

Soil
Microplastics
Polylactide
Carbon radiolabeling

ABSTRACT

The transition towards sustainable and circular materials is imperative to counter the continued rise in global plastic products derived from fossil fuel sources. Bioplastics derived from renewable resources, such as polylactide (PLA), are promising alternatives because they decrease their dependence on such non-renewable carbon-based resource. However, uncertainties remain regarding their recyclability and degradation under natural soil conditions. Assessing bioplastic degradation in soils is challenging due to the complex interplay between polymer properties and variable environmental factors, which limits the effectiveness of conventional analytical methods. Radiocarbon (^{14}C) labeling provides a robust approach to directly quantify polymer mineralization and trace residual material in such environmental matrices.

The degradation of ^{14}C -labeled Polylactide (PLA) was investigated over 24 weeks in two German soils: (i) a loamy sand (Gleyic Cambisol) from Kaldenkirchen-Hülst and (ii) a silt loam (Orthic Luvisol) from Merzenhausen under controlled laboratory conditions. The influence of temperature ($6 \pm 1^\circ\text{C}$, $29.5 \pm 0.5^\circ\text{C}$, $58 \pm 3^\circ\text{C}$, and ambient), soil moisture (70 % of water-holding capacity of soil), and microcrystalline cellulose (MCC) addition was evaluated. Mineralization was quantified by liquid scintillation counting (LSC), while residual PLA was determined using combustion-LSC. In addition, soil pH changes were monitored over time, and structural changes in PLA were tracked by NMR spectroscopy. Based on the results we concluded that temperature is the primary factor controlling PLA degradation, and PLA is not truly biodegradable in soils at temperatures below its glass transition temperature ($T_g \sim 52^\circ\text{C}$).

1. Introduction

Prolonged reliance on petrochemical-derived polymers has led to significant environmental problems, particularly due to poor end-of-life management and the accumulation of non-degradable waste in landfills and, regrettably, in soils [1–3]. The use of bio-based feedstocks as alternatives in plastic production can decrease reliance on fossil resources, ease geopolitical pressures, and reduce climate change impacts linked to greenhouse gas emissions [4]. Bio-based polymers offer the potential to substitute petrochemical plastics across a wide range of applications while delivering new and, in some cases, enhanced material properties. Nevertheless, according to the 2025 European bioplastics market development update, global biobased plastics production capacity represents only about 0.5% of the approximately 431 million tonnes of

plastics produced annually worldwide [5].

The American Society for Testing and Materials (ASTM, 2012) defines biodegradable plastics as materials of natural origin that can be decomposed by microorganisms into carbon dioxide (CO_2), water (H_2O), and biomass under aerobic conditions [6–8]. Similarly, the European Union standard specifies a quantitative criterion for biodegradability, requiring that >90% of a material be converted into CO_2 , water, and mineral substances through biological processes within six months [9, 10]. Notably, these definitions are largely based on degradation under controlled, industrial composting conditions. In natural environments, such optimal conditions are rarely present, meaning that bioplastics, although commonly perceived as fully degradable, often do not undergo complete mineralization. As a result, their degradation is frequently incomplete, leading to fragmentation and the formation of significant

* Corresponding author.

E-mail addresses: s.adeleh@fz-juelich.de (S. Adeleh), tabea.becker@ac.rwth-aachen.de (T. Becker), sonja.herres-pawlis@ac.rwth-aachen.de (S. Herres-Pawlis), r.bol@fz-juelich.de (R. Bol), b.drewes@fz-juelich.de (B. Drewes), t.puetz@fz-juelich.de (T. Pütz).

<https://doi.org/10.1016/j.polyimdegradstab.2026.112348>

Received 15 June 2026; Received in revised form 22 July 2026; Accepted 23 July 2026

Available online 24 July 2026

0141-3910/© 2026 The Author(s). Published by Elsevier Ltd. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

amounts of microplastics (MPs) that can persist in the environment [11].

The pervasive accumulation of plastic debris in terrestrial ecosystems represents a critical threat to global soil health and food security, with plastic concentrations in soil estimated to be 4 to 23 times higher than in marine environments [12]. Unlike aquatic systems where plastic is highly visible, soil acts as a long-term sink for persistent plastics which enter the ground through fragmented agricultural mulch films, contaminated sewage sludge, and atmospheric deposition [13,14]. These materials physically disrupt the soil matrix, altering essential properties such as bulk density and water-holding capacity, which leads to increased evaporation and structural instability [15]. Furthermore, leaching hazardous additives like phthalates and heavy metals while simultaneously adsorbing legacy pesticides and persistent organic pollutants from the surrounding environment [16]. This chemical and physical interference ultimately suppresses the activity of vital soil biota from nitrogen-fixing bacteria to earthworms stifling nutrient cycling and allowing MPs to translocate into the root systems of food crops, thereby integrating synthetic pollutants into the terrestrial food chain [17,18].

Poly(lactide) (PLA) is a biobased and biodegradable polymer derived from lactic acid as its primary building block, which is produced via fermentation of renewable resources such as food crops (e.g., corn) and waste sugars originating from cellulosic materials. This production route combines environmental benefits with desirable mechanical performance. PLA offers notable advantages over petroleum-derived plastics, including substantially lower greenhouse gas emissions and reduced sensitivity to fluctuations in oil prices [19,20].

Initially, the application of PLA was largely restricted to medical uses due to its limited availability; however, advances in technology and the development of methods to produce high-molecular-weight PLA have significantly broadened its range of applications. At present, PLA is predominantly used in the packaging sector [21,22]. In addition, it has gained widespread adoption in other fields, including transportation, electronics, agriculture, textiles, and related industries [23–25].

Although PLA is derived from renewable resources, improper end-of-life management can still contribute to plastic pollution. The common labeling of PLA as “biodegradable” often creates the misleading impression that it rapidly decomposes in natural environments. In reality, the biodegradation of PLA is highly dependent on environmental parameters, leading to inconsistent and sometimes conflicting findings in the literature [26]. Under ambient conditions, such as in natural soils or home composting systems around 20 °C, complete degradation and mineralization of PLA is extremely slow, often requiring well over several years. In contrast, exposure to elevated temperatures around its glass transition temperature ($T_g \sim 50\text{--}65$ °C) can significantly accelerate degradation, shortening the timeframe to approximately 12 weeks [27–29]. With appropriate waste management strategies, including industrial composting and anaerobic digestion, the biodegradability of PLA provides a viable end-of-life pathway that enables complete removal of the material from the environment and supports closure of the carbon cycle [30].

In practice, PLA exhibits significant resistance to microbial degradation in soil and sewage systems characterized by mesophilic temperatures (15–35 °C). Under these ambient conditions, PLA remains in a rigid, glassy state, as the environmental temperature is substantially lower than its T_g . This thermal disparity prevents PLA matrix from achieving sufficient chain mobility, thereby inhibiting the abiotic hydrolysis necessary to render PLA susceptible to microbial enzymatic attack [31]. Consequently, establishing the environmental fate of PLA requires determining the specific duration and extent of exposure to non-mesophilic conditions required to initiate degradation.

PLA primarily enters soil through agricultural applications, such as mulch films and seed coatings, as well as through the disposal of “compostable” packaging in landfills, where conditions for complete degradation are rarely met [32]. Once in the soil, PLA undergoes physical fragmentation and partial hydrolysis, forming persistent secondary microplastics [33,34]. Even in the form of MPs, PLA degradation

is strongly governed by external factors: elevated temperature and moisture promote degradation, whereas low temperatures and oxygen-limited conditions substantially slow the PLA degradation. These microplastics can physically alter soil structure by affecting porosity and water retention, while also chemically reducing soil pH through the release of lactic acid [32,33]. Furthermore, these MPs have been shown to disrupt nitrogen dynamics, suppress beneficial microbial diversity, and impair the reproductive efficiency of essential soil fauna such as earthworms [33–35].

In general, MP analysis involves two main stages. The first is the physical characterization of isolated particles to determine their shape, size, and color, using techniques such as stereomicroscopy, fluorescence microscopy, atomic force microscopy (AFM), transmission electron microscopy (TEM), and scanning electron microscopy (SEM). The second is the chemical characterization to confirm the plastic identity of the detected particles, employing methods such as Raman spectroscopy, Fourier-transform infrared (FT-IR) spectroscopy, and thermal analysis [36,37]. While, these techniques are not capable of directly quantifying MPs degradation at environmentally relevant concentrations in complex matrices like soil, particularly when degradation occurs slowly.

The absence of standardized protocols for MPs recovery from soil often undermines the reliability of environmental research. The lack of harmonization prevents reliable comparison across studies and has caused substantial variability in results reported by laboratories in interlaboratory trials [38].

In this context, sensitive carbon-14 radiolabeling approaches offer a unique advantage, as they represent the only reliable means to directly quantify the degradation, uptake, or environmental distribution of labeled polymer fragments or additives under natural conditions. So, this technique can be used for degradation studies when standard methods are insufficient [39]. As radioactive carbon can be incorporated at specific positions within target molecules in place of stable carbon without modifying the polymer's structure or physicochemical properties, ^{14}C represents a uniquely powerful tracer for environmental fate studies of polymers [40].

Previous literature consistently characterizes PLA degradation as a thermally activated, multifactorial process, where ambient soil temperatures, often below T_g threshold, result in negligible mass loss and structural persistence [41–43]. Traditional soil burial assessments have relied heavily on descriptive physical and mechanical metrics, such as weight loss, tensile strength reduction, Differential Scanning Calorimetry (DSC), FTIR, X-ray diffraction (XRD) analysis, and SEM visualization [44–47], which often conflate PLA fragmentation with true microbial mineralization. While advanced investigations have utilized thermal analysis techniques to correlate changes in calorimetric and viscoelastic properties with molecular weight degradation [48,49], and explored the ‘priming effect’, where soil organic carbon turnover is either accelerated or inhibited by the presence of MPs via microbial co-metabolism [50], these methods often provide only indirect evidence of the degradation stage.

In this study, a carbon-14 radiolabeling approach was employed to directly quantify the degradation of micro- ^{14}C -PLA over a 24-week period in a loamy sand (Gleyic Cambisol) and a silt loam (Orthic Luvisol) soils collected from Kaldenkirchen-Hülst and Merzenhausen (Germany). Experiments were conducted under controlled conditions, including varying temperatures (6 ± 1 °C, 29.5 ± 0.5 °C, 58 ± 3 °C, and ambient soil conditions), a consistent moisture level (70% of water-holding capacity), and the addition of microcrystalline cellulose.

By applying ^{14}C labeling, this study overcomes the limitations of predominantly descriptive methodologies and enables direct quantification of true mineralization. The systematic comparison of soil textures, combined with the use of microcrystalline cellulose (MCC) to standardize microbial substrate availability, allows for a clear distinction between physical fragmentation and actual metabolic carbon turnover. As a result, this work provides a more robust and mechanistically grounded kinetic framework than conventional approaches based

solely on visual or mechanical indicators.

2. Experimental section

2.1. Materials and instruments

Filter paper (whatman), LSC cocktails: Oxysolve C-400 (Zinsser analytical) and Insta-gel PLus (PerkinElmer), Methanol (VWR Chemicals, LC-MS / UHPLC grade, $\geq 99.9\%$ purity), Chloroform (VWR Chemicals, General Purpose Reagent 99.0%–99.6% purity), Microcrystalline cellulose (MCC) (Carl Roth), Sodium hydroxide pellets (VWR Chemicals, Purity: 99.5%), Filtrated rainwater.

Uniformly radiolabeled micro Polylactide (^{14}C -L-PLA; particle size 20–500 μm ; $M_w = 41,700$ g/mol; specific radioactivity = 10.3 kBq/g; glass transition temperature (T_g) = 52 $^{\circ}\text{C}$; melting point (T_m) = 137 $^{\circ}\text{C}$) was used in this study. The details for micro ^{14}C -L-PLA synthesis can be found in earlier work [51].

Liquid scintillation counter (LSC) (PerkinElmer Tri-Carb 3110 TR, Software: QuantaSmart 4.02), Oxidizer (Hidex 600 OX), Data logger temperature (testo 176 T2, software testo comfort software basic), Oven (Binder). Thermostat cabinet (Lovibond, Type: 200,071), Laboratory refrigerator (VWR, Type: 230,874), pH meter (WTW Multi 197i, with probe: SenTix® 41), Drum hoop mixer (J. Engelsmann AG, JEL RRM Mini-II), Control shaker (IKA® KS 260 Basic), Rotary shaker (Gerhardt, shaker (S12), Traceable® digital thermometer (VWR, Accuracy: ± 1 $^{\circ}\text{C}$), Analytical balance (Mettler Toledo, Model: Mettler PC440, Mettler PM4800 DeltaRange, Satorius, Model: BP211D).

2.2. ^1H NMR spectroscopy

^1H NMR spectra were recorded on a Bruker Avance NEO (400 MHz, 9.4 T) software: TopSpin 4.0.2 nuclear resonance spectrometer at 298 K. Resonances were referenced to the residual solvent resonance, relative to TMS (^1H -NMR: CDCl_3 ; $\delta = 7.26$ ppm). Data in the ^1H NMR spectra are stated as follows: chemical shift (δ in ppm) (multiplicity, coupling constant (J in Hz), integration). To describe the multiplicity of the signals the following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or combinations thereof. For visualization and examination of the NMR spectra the software MestReNova (Version 15.0.1–35,756) from Mestrelab Research was used.

2.3. Soil texture and type

Loamy sand and silt loam soils were collected from agricultural field sites at Kaldenkirchen-Hülst (KAL) and Merzenhausen (MER) in North Rhine-Westphalia, Germany. The KAL soil, developed from floodplain loam and terrace sands, is classified as a Gleyic Cambisol with a loamy sand texture [52,53]. In contrast, the MER soil has a silt loam texture and is classified as an Orthic Luvisol [54]. These sites are predominantly used for the cultivation of sugar beet, winter wheat, and winter barley, with potatoes grown occasionally. Samples were obtained from a single soil pit at depths of 0–33 cm (KAL) and 0–35 cm (MER). After collection, the soils were sieved through a 2 mm mesh to remove coarse fragments (> 2 mm), air-dried, and stored in sealed, dark containers under cool conditions until further use. Detailed soil characteristics are provided in Table S1 [56,57].

2.4. Soil pH determination

The method used by Forster [58] was adapted to measure pH of soil before and after degradation of PLA in the soils at week 4, 8, 12, and 24. Air-dried and sieved (< 2 mm) soil samples were suspended in distilled water at a 1:2.5 (w/v) ratio, allowed to equilibrate at room temperature for 1 h, briefly stirred, and the pH of the supernatant was measured using a calibrated pH meter. (Table S1).

2.5. Water holding capacity (WHC) and water content (WC) determination in the soils

Water content was determined by weighing 10 g soil samples ($n = 5$) placed on pre-dried watch glasses to obtain the initial weight (w_{ini}). Samples were oven-dried at 105 $^{\circ}\text{C}$ for 24 h to constant weight (w_{final}), and water content was calculated according to Eq. (1) [59].

$$\text{WC}\% = \frac{w_{\text{ini}} - w_{\text{final}}}{w_{\text{ini}}} * 100 \quad (1)$$

The water holding capacity (WHC) of the soil was determined using a modified Forster protocol [58]. With 100 g of air-dried soil ($n = 5$) was placed in a funnel lined with filter paper, gradually saturated with distilled water, and left to stand for several hours with the funnel covered by aluminum foil to minimize evaporation. After excess water had drained, the saturated soil was weighed (w_{sat}). The sample was then oven-dried at 105 $^{\circ}\text{C}$ for 24 h to constant weight to obtain the dry mass (w_{dry}). WHC was calculated according to Eq. (2).

$$\text{WHC} (\%) = \frac{w_{\text{sat}} - w_{\text{dry}}}{w_{\text{sat}}} * 100 \quad (2)$$

The final calculation for KAL and MER soil is summarized in Table S1. The WC of each flask was set to 70% of the WHC of soil. Accordingly, 30 mL of rainwater was added to 100 g of the KAL soil and 35 mL to the MER soil, based on their initial moisture contents and WHC values.

2.6. Flask preparation and ^{14}C trap analysis

Micro ^{14}C -PLA particles were homogenized by a rotary shaker for 6 h then by adding 2 g of ^{14}C -PLA per 100 g of soil and thoroughly mixed for 4 h to achieve homogeneity. This amount of sample was intentionally applied to optimize isotopic signal resolution for the $^{14}\text{CO}_2$ traps, ensuring robust measurement sensitivity, particularly during low-temperature treatments where mineralization rates were expected to be low. While these concentrated carbon inputs interact with local soil parameters, such as micro-porosity, oxygen diffusion pathways, and carbon-to-nitrogen (C:N) dynamics, they ensure a reliable analytical response throughout the varying conditions of the study. Therefore, the observed degradation kinetics and associated geochemical responses should be interpreted within the context of these experimental loading conditions, recognizing that the results may be concentration-dependent.

A total of 102 g of this mixture was placed in each flask, and the soil moisture was adjusted to 70% of WHC of each soil (KAL and MER) using rainwater. Each week, the moisture deficit of the flasks was corrected by adding rainwater based on weight calculations. To examine the effect of temperature on degradation, three replicates of each soil sample were incubated at 6 ± 1 $^{\circ}\text{C}$, ambient, 29.5 ± 0.5 $^{\circ}\text{C}$, and 58 ± 3 $^{\circ}\text{C}$. The experiment at 6 ± 1 $^{\circ}\text{C}$ is evidence of decomposition without the presence of temperature factor (just for the experiment without microcrystalline cellulose). The ambient soil temperature provides realistic data on PLA degradation. The 29.5 ± 0.5 $^{\circ}\text{C}$ range aligns with standard conditions for degradation studies [60,61], while the 58 ± 3 $^{\circ}\text{C}$ range offers accelerated conditions.

To support microbial activity in the flasks and evaluate its effect, a parallel experiment was conducted under the same conditions, with the addition of MCC (4 g per 100 g of soil) as a supporting carbon source [60]. To monitor mineralization via $^{14}\text{CO}_2$ production, each flask was equipped with 10 mL of 2 M sodium hydroxide solution (80 g NaOH dissolved in 1 L of water), which served as a $^{14}\text{CO}_2$ trapping solution. Trap replacement was conducted every week for whole samples, then each vial mixed with 10 mL of proper cocktail solution for LSC analysis. Prior to weekly trap replacement and sampling, the heated incubation flasks (58 ± 3 $^{\circ}\text{C}$ and 29.5 ± 0.5 $^{\circ}\text{C}$) were allowed to cool to room temperature with the trapping system remaining sealed. This precaution

was taken to minimize the volatilization and loss of gas-phase compounds that could otherwise occur upon opening the flasks. To reduce chemiluminescence caused by the alkaline trapping solution, the scintillation vials were stored in the dark and cold place for 24–48 h prior to LSC analysis [62].

2.7. Residual PLA analysis in soil

Initial amount of radioactivity was measured by oxidizer for each flask by taking an aliquot of mixed soil and ^{14}C -PLA at the beginning of experiment. To evaluate the residual amount of ^{14}C -PLA in the soil after 4, 8, 12, and 24 weeks, combustion experiment was done for each flask after termination. For this purpose, the remaining soil-polymer mixture was dried under a hood, then approximately 0.5 g of the sample ($n = 5$) was combusted in the oxidizer and analyzed using LSC.

2.8. Sample preparation for ^1H NMR analysis

To evaluate the structural changes of PLA in different soil conditions over time, samples were prepared for ^1H -NMR analysis following this procedure. 10 g of dried soil from each flask were transferred into an appropriate extraction bottle. Subsequently, 10 mL of a 90 mL chloroform and 10 mL methanol (9:1, v/v) mixture was added to each bottle. The bottles were tightly sealed to prevent solvent evaporation and shaken on a shaker for 8 h (rpm: 200). After shaking, the soil samples settled for at least 4 h. Using a pipette, load the liquid extract onto a paper filter, and collect the filtrate in a labeled clean glass jar. Allow the extracted liquid to dry completely in the fume hood. Once the extract is dry, resuspend the sample in 500 μL of deuterated chloroform in a fume hood. Then, use a clean pipette tip to transfer the dissolved sample into a NMR tube [63].

2.9. Calculation of half-life times (DT_{50})

Single first order (SFO) and double first order in parallel (DFOP) were used to calculate half-life times (DT_{50}) using cumulative mineralization percentage at 24 time-intervals. (Eq. (3) and 4) Calculations were conducted using all the kinetic models and χ^2 -values and Akaike information criterion of them were compared [64–66]. Furthermore, visual inspection of the fitted curves was performed for all models. The DFOP model provided the best fit for all soil types at $58 \pm 3^\circ\text{C}$ and for the MER and KAL soils amended with MCC under ambient temperature, whereas the remaining datasets were best described by the SFO model. The DT_{50} for all conditions is summarized in Table S2.

$$c_t = c_0 \cdot e^{-k \cdot t} \quad (3)$$

C_t =remaining percentage at time t ; C_0 =initial concentration (100%). at $t = 0$; k =the SFO rate constant (day^{-1}) and t =time.

$$c_t = c_0 [(g)e^{-k_1 t} + (1 - g)e^{-k_2 t}] \quad (4)$$

c_t =remaining percentage at time t ; C_0 =initial concentration (100%). at $t = 0$; k_1 = rate constant for the fast compartment (day^{-1}); k_2 = rate constant for the slow compartment (day^{-1}) and t =time (days).

2.10. Determination of activation energy (E_a), Q_{10} temperature coefficients, and Eyring activation parameters for ^{14}C -PLA mineralization

Kinetic parameters were determined using the two controlled incubation temperatures ($29.5 \pm 0.5^\circ\text{C}$ and $58 \pm 3^\circ\text{C}$), where measurable PLA mineralization occurred. Because only two constant temperature treatments were suitable for kinetic analysis, the activation energy (E_a) was estimated using a two-point Arrhenius calculation (Eq. (5)). The Q_{10} temperature coefficient was calculated to evaluate the temperature sensitivity of PLA mineralization in the soil matrices (Eq. (6)) [67]. (Arrhenius plots are shown in Figures S1–S4). The $6 \pm 1^\circ\text{C}$ treatment

was excluded because no measurable mineralization was detected, while the ambient treatment was excluded because fluctuating temperatures (0 – 50°C) could not be used for a standard Arrhenius analysis. To further characterize the thermodynamic requirements of PLA mineralization, transition state theory was applied using the Eyring-Polanyi equation to determine the enthalpy of activation ($\Delta H^\ddagger$; Eq. (7)) and entropy of activation ($\Delta S^\ddagger$; Eq. (8)) [68,69]. The resulting E_a , Q_{10} , $\Delta H^\ddagger$, and $\Delta S^\ddagger$ values for the different soil types are summarized in Table 1.

$$E_a = \frac{R \cdot \ln\left(\frac{k_2}{k_1}\right)}{\left(\frac{1}{T_1} - \frac{1}{T_2}\right)} \quad (5)$$

$$Q_{10} = \left(\frac{k_2}{k_1}\right)^{\frac{10}{T_2 - T_1}} \quad (6)$$

$$\Delta H^\ddagger = E_a - RT_{\text{avg}} \quad (7)$$

$$\Delta S^\ddagger = R \left[\ln\left(\frac{k_1}{T_1}\right) - \ln\left(\frac{k_2}{T_2}\right) \right] + \frac{\Delta H^\ddagger}{T_1} \quad (8)$$

K_1 and K_2 represent the mineralization rate at 29.5°C (T_1) and 58°C (T_2); T : temperature (All temperatures were converted to the Kelvin scale where T_{avg} is the mean of T_1 and T_2); R : $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$; k_B : $1.381 \times 10^{-23} \text{ JK}^{-1}$; h : $6.826 \times 10^{-34} \text{ Js}$

2.11. Statistical analyses

Data are reported as mean ($n = 3$) $\pm$ standard deviation (SD) unless otherwise stated. A three-way repeated measures ANOVA with Tukey's HSD test was performed in jamovi (v2.7.13) to assess the effects of temperature, time, and soil type, with significance set at $p < 0.05$. Additionally, to isolate localized matrix mechanics and resolve high-order interactions, separate two-way repeated measures ANOVAs followed by Tukey's HSD post-hoc pairwise comparisons were conducted independently for each thermal regime and soil type.

3. Results

3.1. PLA degradation at four different temperature scenarios

Increasing the incubation temperature to a thermophilic level ($58 \pm 3^\circ\text{C}$) strongly affected the mineralization of micro- ^{14}C -PLA. Unlike at lower temperatures, no long lag phase was observed, and the loss of ^{14}C -PLA increased rapidly (Fig. 1a). Mineralization started within the first week in all treatments. The results showed higher degradation in the MER soil, both with and without MCC, reaching about 38% cumulative mineralization after 24 weeks. In contrast, the unamended KAL soil showed the lowest degradation, with the mineralization rate slowing down early and reaching only 24% by week 24. The KAL+MCC treatment showed intermediate mineralization, reaching 35% at the end of the incubation. These results suggest that thermophilic conditions, which are above the glass transition temperature (T_g) of PLA and promote hydrolysis, enhance PLA degradation. The highest degradation was observed in MER soil, indicating that the combination of high temperature and favourable soil properties, including its silt loam texture, higher water-holding capacity, nutrient retention, and fertility,

Table 1
 E_a and Q_{10} values in different soil types.

	KAL	MER	KAL+MCC	MER+MCC
E_a (kJ mol^{-1})	295	246	278	294
Q_{10}	34	19	28	30
$\Delta H^\ddagger$ (kJ/mol)	292.4	242.9	275.1	290.8
$\Delta S^\ddagger$ (J/mol)	542.1	392.7	495.2	540.3

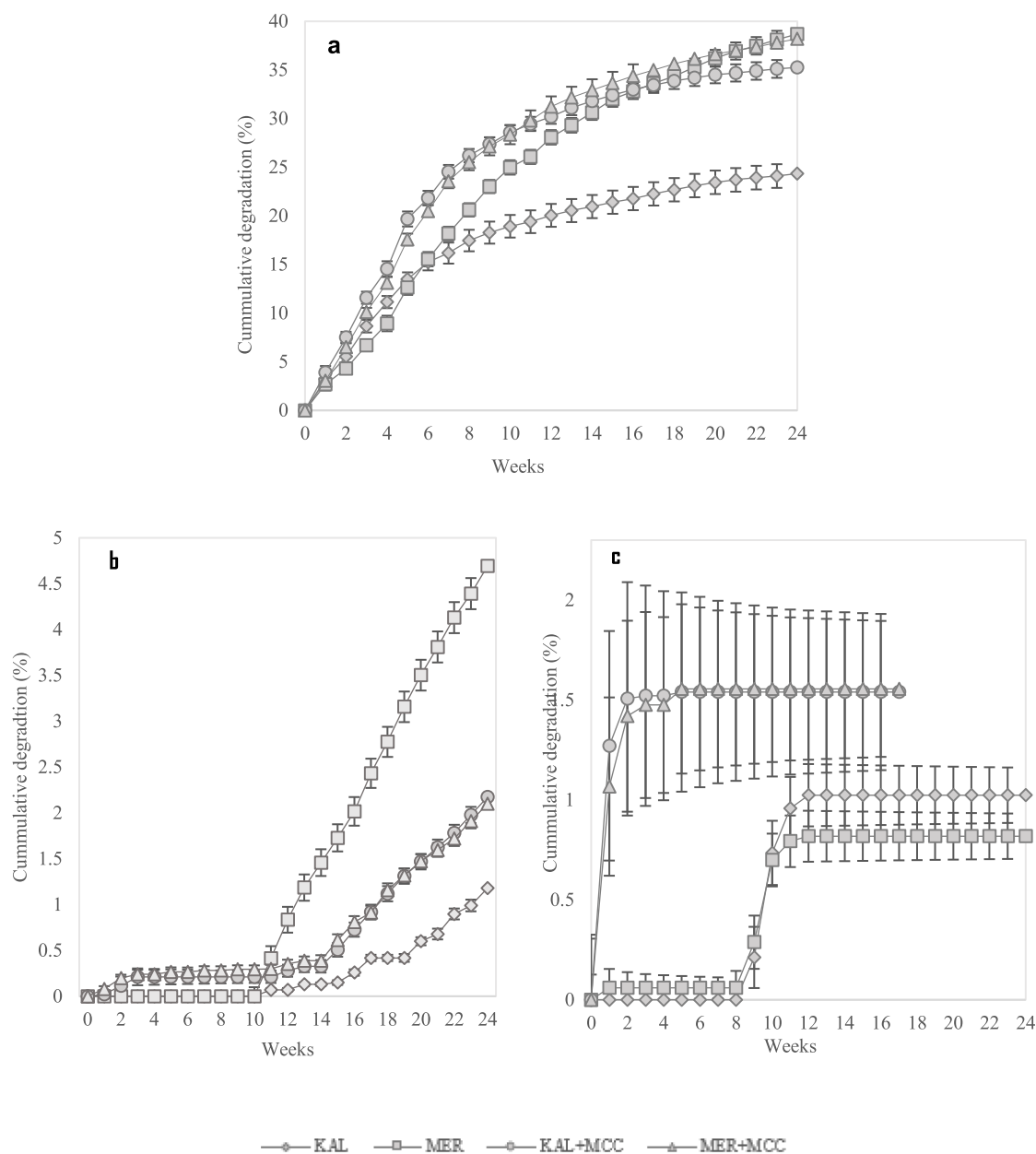


Fig. 1. Cumulative mineralization (%) of micro-¹⁴C-PLA in KAL and MER soils, with and without MCC amendment, over a 24-week incubation under varying temperature conditions: (a) 58 ± 3 °C, (b) 29.5 ± 0.5 °C, and (c) ambient soil temperature. (calculated based on the specific radioactivity of ¹⁴C-PLA).

supported the breakdown and mineralization of the polymer carbon [9, 70–72].

The soil combustion analysis supported the CO₂ mineralization results and showed a strong decrease in the amount of remaining PLA over time in all treatments (Fig. 2 and Figure S5). A rapid loss of 23–27% of the initial PLA mass was observed during the first four weeks of incubation at high temperature in all soils. Consistent with its high mineralization activity, the unamended MER soil contained only $19 \pm 2\%$ of the initial PLA after 24 weeks. In comparison, $49 \pm 1\%$ of the initial PLA remained in the unamended KAL soil. In the cellulose-amended treatments, the remaining PLA was $35 \pm 1\%$ in MER+MCC and $51 \pm 1\%$ in KAL+MCC at the end of the experiment.

Although the unamended MER and MER+MCC treatments reached a similar final cumulative mineralization value (about 38%), less PLA residue remained in the unamended MER soil. This suggests differences in the fate of PLA-derived carbon between the two treatments. One

possible explanation is that the addition of MCC promoted the incorporation of PLA-derived carbon into microbial biomass instead of its immediate conversion to CO₂. Another possibility is that the high microbial activity in MER soil created small oxygen-limited zones. Under such conditions, part of the carbon may have been converted to methane (CH₄), which was not measured in this study. Further research, including gas chromatography measurements [73] and microbial biomass analyses [74], is needed to better understand the distribution of PLA-derived carbon among CO₂, microbial biomass, and other carbon products.

In addition to the loss of PLA mass, the high incubation temperature caused major changes in the structure of the polymer. From week 8 onward, individual PLA microparticles could no longer be observed in the soils. The particles became completely opaque, indicating that the polymer had undergone substantial physical, mechanical, and chemical changes together with microbial degradation and assimilation [75,76].

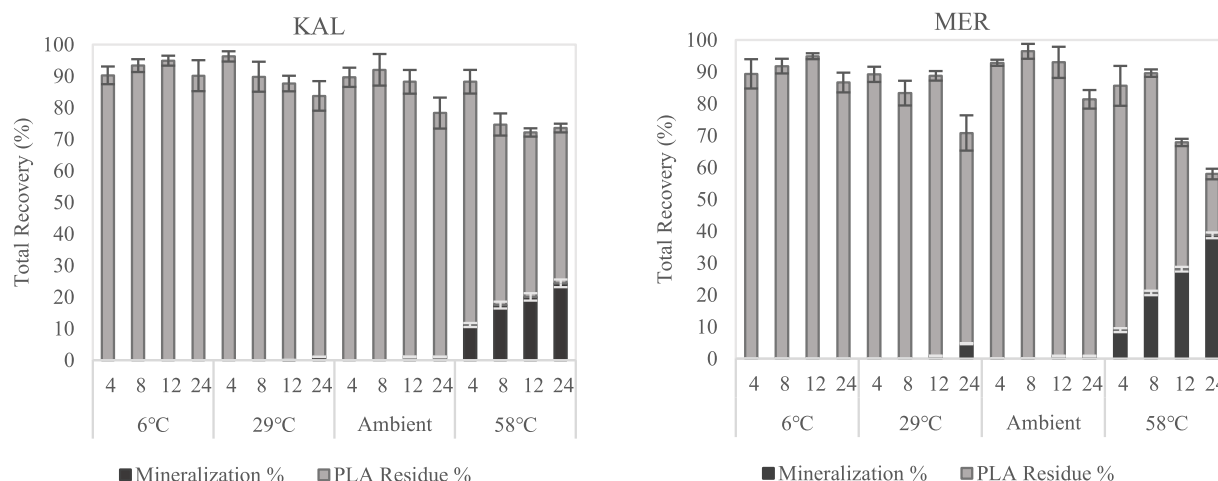


Fig. 2. Mass balance of micro ^{14}C -PLA degradation in KAL and MER soils across different temperatures. (calculated based on the specific radioactivity of ^{14}C -PLA).

At an incubation temperature of 29.5 ± 0.5 °C, the mineralization of ^{14}C -PLA was strongly affected by soil type and the addition of MCC (Fig. 1b). All treatments showed a clear lag phase. During the first 11 weeks, PLA degradation was very limited, and cumulative mineralization remained below 0.4%. However, soils amended with MCC showed slightly higher mineralization during this early period. After week 11, mineralization increased, especially in the MER soil. The cumulative mineralization in MER increased almost linearly at a rate of 0.33% per week ($R^2 = 0.997$), which was about five times higher than in the KAL soil. As a result, MER reached a final cumulative mineralization of 4.7% after 24 weeks, while KAL reached only 1.2%.

The MCC-amended treatments (KAL+MCC and MER+MCC) showed similar mineralization patterns after week 11 and both reached a final mineralization of about 2.2%. These results suggest that MCC may slightly enhance PLA degradation during the early stages of incubation. Over time, MCC appeared to reduce the differences between the two soils, leading to more similar microbial activity and degradation rates and reducing the naturally higher degradation observed in MER soil.

To confirm the mineralization results, soil combustion analysis was performed to measure the amount of PLA remaining in the soil over time (Fig. 2 and Figure S5). The radiocarbon measurements in the soil supported the mineralization data and showed a progressive decrease in PLA content during the experiment. In agreement with the low mineralization rates expected below the glass transition temperature ($T_g \sim 50$ – 65 °C), most of the PLA remained in the KAL, KAL+MCC, and MER+MCC treatments after 24 weeks, with residual PLA contents of $83 \pm 5\%$, $87 \pm 4\%$, and $81 \pm 1\%$, respectively. Consistent with its higher CO_2 production, the unamended MER soil showed the greatest PLA degradation, with only $66 \pm 6\%$ of the initial PLA remaining at the end of the study. Although PLA degradation is substantially accelerated above T_g , slow hydrolytic chain scission can still occur below T_g during prolonged incubation, producing low-molecular-weight oligomers that are subsequently assimilated and mineralized by soil microorganisms. The higher mineralization observed in MER compared with KAL further indicates that soil-specific factors contributed to PLA degradation under mesophilic conditions. Therefore, the measurable mineralization observed at 29.5 ± 0.5 °C was likely the result of prolonged incubation, slow hydrolysis below T_g , and soil-specific conditions rather than thermal softening of the polymer. The close agreement between the higher CO_2 production and the lower residual PLA content confirms that the observed CO_2 evolution resulted from PLA degradation.

Soil temperature and its fluctuations played an important role in controlling the mineralization of ^{14}C -PLA in the ambient soil treatment (Fig. 1c). In all treatments, the highest degradation rates were observed during the warmest period of the experiment, from 6th August to 4th September. During this 30-day period, the average daily soil

temperature was 24.8 ± 2.5 °C in KAL and 23.9 ± 2.1 °C in MER. To estimate the total amount of heat available for degradation, cumulative temperature sums were calculated using a base temperature of 0 °C ($T_{\text{base}} = 0$ °C). During this warm period, KAL accumulated a higher temperature sum (745.3 °C·d) than MER (718.5 °C·d), indicating a greater thermal input that could support the initial breakdown of PLA. Even so, the average temperatures do not reflect the large daily temperature fluctuations. Maximum soil temperatures reached 49.6 °C in KAL and 46.5 °C in MER, while minimum temperatures dropped to 1.3 °C and 0.6 °C, respectively (Figure S5). These results show that the soils experienced substantial temperature changes between day and night during the experiment. (Figure S6)

Among the unamended soils, KAL showed higher cumulative $^{14}\text{CO}_2$ mineralization of ^{14}C -PLA than MER. This difference is likely related to the lower heat capacity of KAL soil, which allowed it to warm up and cool down more quickly. As a result, microbial activity may have increased more rapidly during warm periods. However, ambient temperature conditions alone were not sufficient to support substantial long-term PLA degradation. After 24 weeks, cumulative mineralization remained below 1.0% in KAL and below 0.8% in MER, despite both soils receiving a similar total amount of thermal energy over the course of the experiment (3181.1 °C·d for KAL and 3123.7 °C·d for MER) (Fig. 1c). The small difference in cumulative temperature sums (about 1.8%) suggests that total heat input was not the main factor controlling PLA degradation under ambient conditions. Instead, the higher daily temperature peaks observed in KAL were likely more important, as they may have helped overcome the energy barrier required to initiate polymer breakdown.

After 6th September, CO_2 production decreased rapidly as the maximum daily soil temperatures fell below 30 °C and the average daily temperature dropped to 12 ± 5 °C. This suggests that PLA degradation under ambient conditions requires a minimum temperature threshold. Below this threshold, microbial activity and enzymatic hydrolysis were strongly reduced. As a result, most of the radiolabeled PLA remained undegraded in the soil by the end of the experiment, with $77 \pm 5\%$ remaining in KAL and $81 \pm 3\%$ remaining in MER (Fig. 2).

To determine whether microbial activity could be enhanced, MCC was added as an additional carbon source to stimulate co-metabolism. In the ambient-temperature experiment, MCC was added after an 8-week delay (from 7th August to 9th December 2025), and the treatments were monitored for 17 weeks. Due to the colder weather during this period (average temperature 12 ± 5 °C), measurements for these treatments were stopped after week 17. The seasonal decrease in temperature greatly reduced the available thermal energy, resulting in cumulative temperature sums of only 1907.1 °C·d for KAL+MCC and 1858.5 °C·d for MER+MCC during the 17-week incubation period.

The addition of MCC significantly altered the degradation behavior of PLA through co-metabolism. Following MCC amendment, cumulative $^{14}\text{CO}_2$ production increased rapidly, reaching 1.3% mineralization in KAL and 1.1% in MER within the first week (Fig. 1c). This pronounced initial response suggests a substantial co-metabolic enhancement driven by the readily available cellulose, which stimulated microbial activity and accelerated PLA mineralization. The increase in microbial activity occurred during a period of relatively high outdoor temperatures, with average daily temperatures of $27.6 \pm 10^\circ\text{C}$ in KAL and $26.6 \pm 9^\circ\text{C}$ in MER. These were similar temperature conditions to those under which PLA degradation had first started in both soils. This observation suggests that both biological stimulation by MCC and elevated temperatures are important for initiating PLA degradation under ambient conditions. Together, they help overcome the high energy barrier required for PLA breakdown. Despite the increase in microbial activity caused by MCC, overall PLA mineralization remained low. Cumulative CO_2 production stayed below 1.6% of the initial $\sim 2\text{ g}$ of ^{14}C -PLA. Residual PLA analysis at week 17 showed that most of the polymer remained intact and visible in the soil, with $85 \pm 2\%$ remaining in KAL+MCC and 87% remaining in MER+MCC (Figure S5).

During the 24-week incubation period, no measurable CO_2 production was detected in either the MER or KAL soils incubated at $6 \pm 1^\circ\text{C}$. As a result, most of the PLA remained unchanged throughout the experiment, with about $88 \pm 4\%$ of the original polymer still present after 24 weeks (Fig. 2). The micro-PLA particles were still clearly visible at the end of the incubation. While, soil combustion analysis showed a degree of degradation, corresponding to $10 \pm 7\%$ in KAL and $13 \pm 3\%$ in MER. The observed values likely reflect a combination of analytical uncertainty and sample handling effects. For instance, the analysis of small soil subsamples (0.5 g) by oxidizer–LSC may introduce variability due to inherent spatial heterogeneity within the soil matrix, while post-incubation air-drying under a fume hood may have facilitated the loss of volatile or semi-volatile ^{14}C -labeled degradation products. Nevertheless, a limited contribution from low-temperature hydrolytic weathering or moisture-induced surface chain scission cannot be completely excluded under the experimental conditions employed.

The two-way repeated measures ANOVA analyses datasets across the 4, 8, 12, and 24-week intervals, successfully demonstrated that temperature serves as the primary driver governing both PLA residual analysis and cumulative mineralization, shifting the underlying degradation kinetics from a matrix-dependent regime to a thermally dominated one.

A two-way repeated measures ANOVA at $58 \pm 3^\circ\text{C}$ showed that thermophilic conditions promote rapid, heat-driven degradation, which becomes the dominant process and ultimately results in similar outcomes across all soil types. For residual polymer content, highly significant main effects for time ($F_{3,15} = 166.12$, $p < 0.001$) and soil type ($F_{3,5} = 11.6$, $p = 0.011$) were observed alongside a significant interaction ($F_{9,15} = 2.91$, $p = 0.033$), confirming that degradation rates fluctuated between soils during intermediate weeks before reaching to an identical statistical endpoint by week 24 ($p > 0.05$). While the KAL soil matrix visually displays the lowest cumulative mineralization percentage, both the soil main effect ($p = 0.133$) and the time $\times$ soil interaction ($F_{9,24} = 2.15$, $p = 0.065$) were statistically non-significant. This lack of statistical significance proves that the MER and KAL soils with and without MCC followed highly uniform, parallel carbon turnover pathways, and terminal post-hoc analysis at week 24 confirmed that the visible gaps on the chart do not reflect true statistical differences ($p > 0.05$).

Mesophilic conditions ($29.5 \pm 0.5^\circ\text{C}$) unlocks distinct biological and structural pathways, establishing highly significant time $\times$ soil interactions for both residue PLA ($F_{9,24} = 4.44$, $p = 0.002$) and cumulative mineralization ($F_{9,24} = 9.42$, $p < 0.001$). This strong mineralization interaction indicates that degradation rates differed greatly between soils over time. Some soils showed a rapid increase in CO_2 production during the early stages of incubation, while others exhibited a slower response and entered active mineralization later. This pattern was

mainly driven by the unamended MER soil, which experienced an early increase in mineralization and ultimately achieved significantly higher PLA mineralization than the other treatments.

In isolated comparisons of the unamended soils at ambient soil temperature, the soil effect was highly significant ($F_{1,4} = 113.00$, $p < 0.001$), but the addition of a MCC co-substrate effectively neutralized this variance ($p = 0.523$), forcing both amended matrices to progress along highly uniform, residual PLA content degradation ($p = 0.601$) and parallel mineralization ($p = 0.977$) pathways.

At $6 \pm 1^\circ\text{C}$, low temperatures strongly limit microbial activity, resulting in no significant effects of time ($p = 0.333$) or soil type ($p = 0.906$). The interaction between time and soil type was also weak ($p = 0.042$) in the unamended soils.

3.2. Mass balance accounting

The mass balance of ^{14}C -PLA was determined from the cumulative recovery of ^{14}C in two pools: the mineralized fraction, quantified as evolved $^{14}\text{CO}_2$, and the total residual radioactivity (TRR), measured by direct combustion of the soil matrix (Fig. 2 and Figure S5). This approach enabled comprehensive accounting of polymer-derived carbon, including the parent polymer, intermediate metabolites, and non-extractable (bound) residues. The inverse relationship between increasing $^{14}\text{CO}_2$ evolution and declining TRR demonstrates the progressive mineralization of PLA-derived carbon.

However, incomplete ^{14}C recovery was observed, particularly at the elevated incubation temperature ($58 \pm 3^\circ\text{C}$). This is primarily attributed to the heterogeneous distribution of residual polymer within the soil matrix, as analysis of small soil subsamples (0.5 g) can introduce variability when undegraded polymer fragments and localized degradation products are unevenly distributed. In addition, part of the unrecovered ^{14}C may have been present as volatile or low-molecular-weight degradation products. Although precautions were taken to minimize the loss of volatile compounds during sampling by cooling the sealed incubation flasks to room temperature before opening them, some losses likely occurred during post-incubation processing, when the residual soil–PLA mixture was air-dried under a fume hood without trapping volatile compounds. Consequently, evaporation of volatile or semi-volatile ^{14}C -labeled degradation products during drying likely contributed to the incomplete mass balance and represents a limitation of the experimental approach.

3.3. Temporal soil pH values

The dynamic shifts in soil pH during the 24 weeks incubation period reflect a complex interplay between PLA degradation kinetics, soil buffering capacity, and the cumulative effect of irrigation rainwater ($\text{pH}=9.82$). The initial decline in pH is attributed to the hydrolysis of PLA and the subsequent release of lactic acid ($\text{pH}=2.27$ for 10% solution) and carboxyl groups into the soil. The decrease in pH at high temperature is more pronounced due to the increased degradation of PLA overtime (Fig. 3 and Figure S7).

3.4. ^1H NMR characterization of PLA degradation in soil

The ^1H NMR spectra of PLA extracted from soil were analyzed to monitor PLA structural changes under varying soil conditions over time; the spectra exhibit two characteristic signals associated with the PLA backbone: a doublet at 1.58 ppm corresponding to the methyl protons ($-\text{CH}_3$) and a quartet at 5.16 ppm corresponding to the methine proton ($-\text{CH}-$) (Figure S8) [51]. ^1H NMR analysis of the incubation samples at $6 \pm 1^\circ\text{C}$, ambient temperature, and $29.5 \pm 0.5^\circ\text{C}$ showed no changes over a period of 24 weeks in either of the two soil types with and without MCC (Figure S9–S18). In contrast, the ^1H NMR spectrum of samples incubated at $58 \pm 3^\circ\text{C}$ revealed that, in MER soil, the two characteristic PLA peaks were absent at week 24 (Fig. 4a). In KAL soil, the intensities of

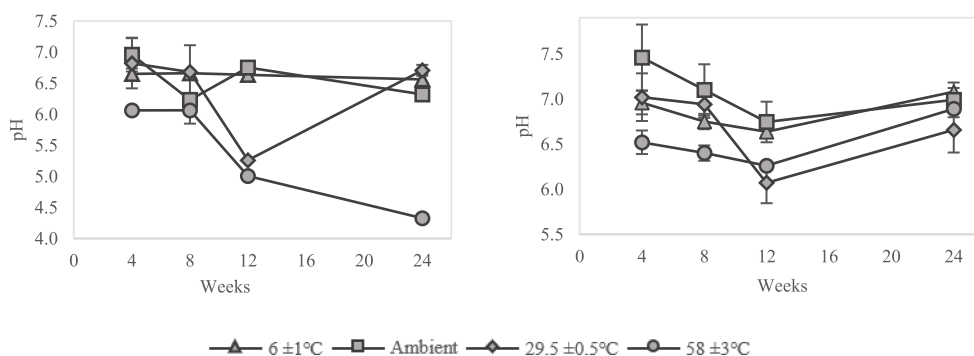


Fig. 3. Variation of pH over time for KAL (left) and MER (right) soil at different temperatures.

these peaks were significantly reduced, and evidence of lactic acid presence was observed, indicated by the appearance of a corresponding peak a quartet at $\delta = 4.1\text{--}4.3$ ppm assigned to the methine proton ($-\text{CH}-$) and a doublet at $1.2\text{--}1.4$ ppm assigned to the methyl protons ($-\text{CH}_3$) at $1.2\text{--}1.4$ ppm (Fig. 4b). The same happened for soils with MCC at this temperature (Figure S19 and S20).

4. Discussion

The soil samples collected from Kaldenkirchen-Hülst and Merzenhausen are not merely site-specific selections but are representative of the broader Central European agricultural belt. This relevance arises from the diverse soil profiles of North Rhine-Westphalia, which are shaped by glacial deposits and the geomorphological evolution of the Rhine valley. Consequently, the contrasting properties of the Orthic Luvisol (silt loam) at Merzenhausen and the Gleyic Cambisol (loamy sand) at Kaldenkirchen-Hülst provide a meaningful framework to investigate how soil characteristics influence PLA degradation processes [70,71]. These inherent soil differences are directly linked to the observed degradation behavior of PLA. While Luvisols are among the most fertile and agriculturally important soils in Central Europe due to their high water-holding capacity and nutrient retention, the sandy Cambisol reflects fluvial conditions with lower retention capacity [77]. Such physicochemical differences are particularly relevant when considering that temperature is the dominant controlling factor in PLA degradation. As widely reported, both hydrolysis rates and microbial activity increase with rising temperature [9], and degradation is significantly enhanced once temperatures exceed the T_g of PLA.

In natural soils, temperatures rarely exceed 30°C [72], meaning that PLA remains below its T_g in a rigid, glassy state, restricting molecular mobility and hydrolytic cleavage. As a result, degradation remains minimal, typically below 10% after six months, with a study reporting only $\sim 4\%$ biodegradation after 200 days [78], and negligible changes even after one year at 25°C [55]. Similar findings by Karamanlioglu et al. confirm the persistence of PLA at ambient soil temperatures [41, 79]. In contrast, under accelerated conditions (active aerobic composting media rich in thermophilic microorganisms, maintained at thermophilic temperatures of $58\text{--}60^\circ\text{C}$ and a slightly alkaline pH of $7.0\text{--}8.0$), PLA can degrade within three months [78,80].

These observations are consistent with the high DT_{50} values obtained in this study, ranging from 73,557 days (over 200 years) for KAL + MCC under soil ambient conditions to 213 days for MER with and without MCC at $58 \pm 3^\circ\text{C}$, confirming that PLA exhibits high persistence under both ambient and $29.5 \pm 0.5^\circ\text{C}$ conditions. Significant degradation occurs only at temperatures $\geq 58^\circ\text{C}$ [75,81] (Table S2), where PLA transitions into a more mobile state, enabling rapid hydrolysis and microbial utilization. The sharp reduction in half-life at $58 \pm 3^\circ\text{C}$ observed here further supports the existence of a critical thermal activation threshold governing degradation kinetics. This temperature dependence is quantitatively supported by Arrhenius-based analyses of activation

energy (E_a) and Q_{10} coefficients, as well as by application of the Eyring-Polanyi framework to determine the enthalpy of activation ($\Delta H^\ddagger$) and entropy of activation ($\Delta S^\ddagger$) for ^{14}C -PLA mineralization. Together, these complementary approaches provide a comprehensive evaluation of the thermodynamic requirements (E_a), the fundamental transition-state dynamics ($\Delta H^\ddagger$ and $\Delta S^\ddagger$), and the temperature-driven acceleration of PLA mineralization (Q_{10}) across different soil environments.

The calculated E_a values ranged from 245.5 to 295.0 kJ/mol , indicating a substantial energy barrier and confirming that the initial degradation of PLA is a highly endothermic, rate-limiting step in soil. Consistent with these findings, the enthalpy of activation ($\Delta H^\ddagger$) ranged from 242.87 to 292.37 kJ/mol , while the strongly positive activation entropies ($\Delta S^\ddagger = +392.6$ to $+542.0\text{ J/(mol}\cdot\text{K)}$) indicated that PLA degradation requires a transition from an ordered polymer structure to a highly disordered activated state. KAL soil exhibited the highest temperature sensitivity ($Q_{10} = 34$), whereas MER soil showed the lowest ($Q_{10} = 19$). The addition of MCC reduced these differences, bringing the Q_{10} values of both soils into a narrower range ($28\text{--}30$) and suggesting a more uniform thermal response across soil types (Table 1). Overall, the high Q_{10} values and Eyring parameters demonstrate that PLA mineralization is highly temperature-dependent, such that even moderate warming may substantially accelerate degradation and carbon turnover in soil.

These mechanistic insights align closely with the statistical analysis of residual PLA content. A three-way mixed-design ANOVA for residual PLA content across four time intervals (4, 8, 12, and 24 weeks) revealed significant main effects of time ($F_{3,63} = 95.88$, $p < 0.001$), temperature ($F_{2,21} = 794.4$, $p < 0.001$), and soil type ($F_{3,21} = 16.4$, $p < 0.001$). Among these factors, temperature exhibited the strongest influence, with 58°C causing a substantial reduction in residual polymer compared to ambient and 29.5°C conditions across all soils. Significant interaction effects, including time $\times$ temperature ($F_{6,63} = 21.67$, $p < 0.001$) and the three-way time $\times$ soil $\times$ temperature interaction ($F_{18,63} = 2.62$, $p = 0.003$), indicate that degradation rates over time depend strongly on the combined effects of soil environment and thermal conditions. Tukey's HSD post hoc analysis further showed that KAL soil with MCC retained the highest residual PLA, whereas MER soil with MCC promoted the most rapid degradation. The findings confirm that PLA degradation is a multifactorial process primarily driven by thermal energy, with $58 \pm 3^\circ\text{C}$ acting as the dominant catalytic condition for PLA breakdown. Although time and soil type significantly affect degradation, their influence is secondary to temperature. The significant three-way interaction highlights that PLA persistence cannot be predicted by soil type alone; rather, specific soil characteristics, such as mineral composition and microbial activity, interact with elevated temperatures to accelerate degradation.

In addition, cumulative mineralization percentage was evaluated over a 24-week period using three-way repeated measures ANOVA. Due to a significant violation of the assumption of sphericity (Mauchly's $W <$

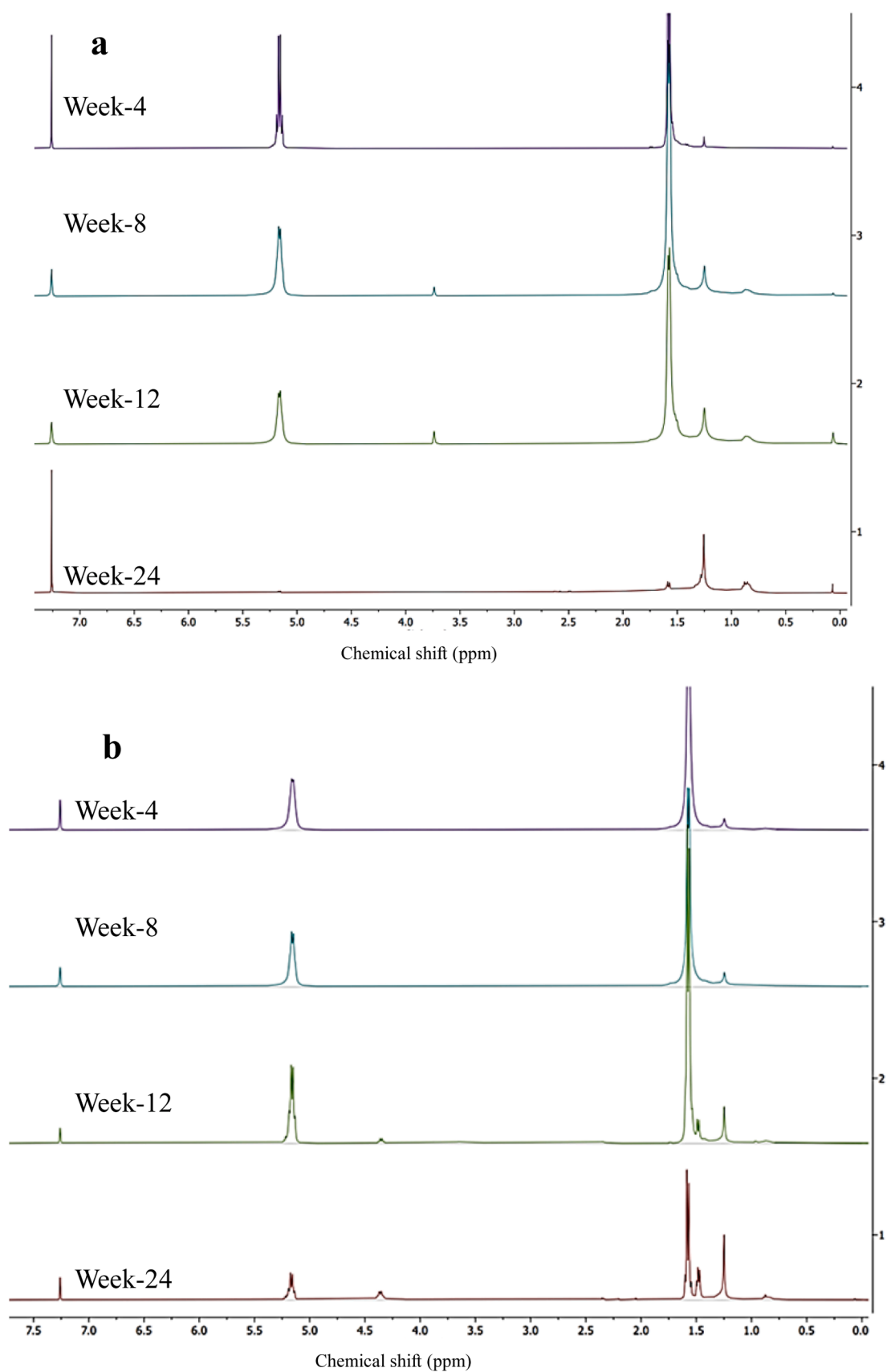


Fig. 4. The ^1H NMR spectra of PLA extracted from MER soil (a) KAL soil (b) overtime at 58 ± 3 °C.

0.001, $p < 0.001$), degrees of freedom were adjusted using Greenhouse-Geisser corrections. The analysis revealed highly significant main effects for time ($F_{1,09, 26,28} = 114.32$, $p < 0.001$) and temperature ($F_{2, 24} = 235.13$, $p < 0.001$), indicating that the total carbon turnover was primarily driven by thermal energy and duration. A significant time $\times$ temperature interaction ($F_{2,19, 52,61} = 91.46$, $p < 0.001$) further confirms that the rate of mineralization was non-uniform across different thermal conditions. Notably, while soil Type was a significant factor in primary PLA degradation, its effect on overall cumulative mineralization was not statistically significant at the 5% level ($F_{3, 24} = 2.62$, $p = 0.074$), and no significant three-way interaction between time $\times$ soil $\times$ temperature was observed ($F_{6,57, 157,56} = 1.75$, $p = 0.144$). These results suggest that while soil environment may influence early-stage fragmentation, long-term mineralization is dominated by temperature-dependent kinetics.

Changes in soil pH provide additional evidence of ongoing degradation. The degradation of PLA MPs in soil significantly decreases soil pH while simultaneously increasing soil carbon content [82]. This pH reduction is attributed to the depolymerization and hydrolysis of PLA, which generate lactic acid as a primary degradation product [41]. During hydrolysis, the formation of carboxyl ($-\text{COOH}$) and hydroxyl ($-\text{OH}$) functional groups further contributes to acidification of the surrounding medium, making pH a commonly used indicator for assessing PLA degradation [83]. The clarity of this trend in KAL soil is due to its inherently low buffering capacity and low cation exchange capacity (CEC), which allows for immediate acidification. In contrast, MER soil showed a more stable pH profile in the early stages, likely due to its superior ability to neutralize acidic byproducts through surface exchange reactions [84,85]. The increase in pH can be attributed to the repeated addition of alkaline rainwater (pH 9.82). As PLA degradation slowed over time, the production of acidic hydrolysis products decreased, while the continuous input of alkaline rainwater gradually neutralized soil acidity, resulting in a recovery of pH. This recovery was more pronounced in MER soil due to its higher buffering capacity.

A closer examination of the temporal pH trends in the four soil systems (KAL, MER, KAL+MCC, and MER+MCC) further explains the patterns shown in Fig. 3 and Figure S7. In KAL soil, pH remained stable at 6 ± 1 °C (6.6–6.7) throughout the 24-week incubation, indicating minimal hydrolytic and microbial activity. At 29.5 ± 0.5 °C, pH decreased from 6.7 to 5.3 by week 12 and then recovered by week 24, suggesting that the acidic products formed during PLA degradation were gradually neutralized by the alkaline rainwater. Under ambient conditions, pH declined from 7.0 to 6.2 during the first 8 weeks and increased to 6.8 by week 12. This recovery coincided with the seasonal decrease in temperature after September 6th, when maximum temperatures fell below 30 °C and average temperatures decreased to approximately 12 ± 5 °C. The lower temperatures slowed PLA degradation, reducing the formation of acidic degradation products, while the continued addition of alkaline rainwater promoted pH recovery. In contrast, the greatest acidification occurred at 58 ± 3 °C, where pH steadily declined from 6.1 at week 4 to 4.3 at week 24, reflecting accelerated PLA hydrolysis, lactic acid production, and the relatively low buffering capacity of KAL soil.

MER soil showed the same temperature-dependent trend but with smaller pH changes and greater recovery. At 6 ± 1 °C and under ambient conditions, pH remained close to neutral (6.6–7.1 and 6.7–7.5, respectively). Even at 58 ± 3 °C, pH decreased only moderately before recovering to 6.9 by week 24, unlike KAL soil. This behavior reflects the higher cation exchange capacity and buffering capacity of MER, which more effectively neutralized the acidic products generated during PLA degradation.

The addition of MCC enhanced the initial decrease in pH without altering the overall trend. In both KAL+MCC and MER+MCC, pH declined rapidly between weeks 4 and 8 under ambient and 29.5 ± 0.5 °C conditions (KAL+MCC: 6.9 to 5.1; MER+MCC: 7.3 to 6.0), indicating increased microbial activity stimulated by the readily degradable carbon source. As microbial activity and PLA degradation slowed, the continued addition of alkaline rainwater gradually neutralized the accumulated

acidity, resulting in partial pH recovery. Under ambient conditions, pH recovered to 6.4 in both KAL+MCC and MER+MCC, while at 29.5 ± 0.5 °C it increased to 6.6 and 6.9, respectively. In contrast, at 58 ± 3 °C, pH continued to decrease throughout the incubation in both KAL+MCC (4.1) and MER+MCC (5.8), indicating that the combined degradation of PLA and MCC exceeded the buffering capacity of the soils. Overall, these results demonstrate that soil pH was controlled by the balance between acid production during PLA degradation and microbial activity and the neutralizing effect of alkaline rainwater, with the response depending on soil buffering capacity, incubation temperature, and the presence of MCC.

The statistical analysis of pH dynamics further reinforces the interconnected nature of these processes. A significant three-way repeated measures ANOVA were conducted to evaluate the effects of soil type, temperature, and time interval on soil pH. The analysis revealed a significant three-way interaction between time, soil, and temperature ($F_{18, 72} = 3.75$, $p < 0.001$), indicating that the temporal stability of pH is contingent upon the specific soil environment and thermal load. Main effects for soil ($F_{3, 24} = 46.65$, $p < 0.001$) and temperature ($F_{2, 24} = 46.67$, $p < 0.001$) were also highly significant, with post-hoc Tukey tests identifying the 58 ± 3 °C treatment as a major driver of alkalinity shifts, particularly in KAL soil ($p < 0.001$). These findings suggest that extreme thermal stress overrides inherent soil buffering capacities, leading to significant fluctuations in pH that are both soil-specific and time-dependent.

Taken together, the findings demonstrate that PLA degradation in soil is a tightly coupled, multifactorial process in which temperature acts as the primary driver, while soil properties modulate the progression of degradation, particularly during the early stages. By integrating kinetic parameters, statistical analysis, and analytical methods, specifically pH monitoring and ^1H NMR spectroscopy, this study provides a coherent framework linking molecular-scale degradation mechanisms with macroscopic soil responses.

The advantages of the ^{14}C -radiolabeling approach are particularly evident when compared with conventional analytical methods. Traditional techniques, including visual inspection, gravimetric mass loss, SEM, FTIR, DSC, and XRD, primarily detect physical fragmentation or changes in polymer chemistry but cannot directly quantify polymer mineralization in complex soil matrices [44–47]. In contrast, the micro ^{14}C -PLA approach directly quantified PLA mineralization through the production of $^{14}\text{CO}_2$ and enabled the tracking of PLA-derived carbon between mineralized CO_2 and soil-bound residues, providing direct insight into the environmental fate of the polymer that cannot be achieved using conventional non-isotopic methods.

Building on these findings, several key research directions emerge to further advance our understanding of the environmental behavior of micro-PLA in soils. To enable robust hazard assessments and cross-study comparability, the development of harmonized protocols for micro-PLA extraction and carbon flux measurement is essential [86–88]. Complementing radiolabeling with advanced analytical techniques, such as SEC/GPC for monitoring molecular weight shifts, [89–91] and SEM/EDX for visualizing microbial pitting and biofilm formation, will bridge the gap between physical degradation patterns and biologically driven CO_2 evolution [32,47,79,92–96]. Furthermore, comprehensive environmental evaluation must extend beyond physical particle recovery to assess the potential toxicological risks of specific additives and polymer formulations [97,98]. Future research requires multi-season field studies to monitor long-term accumulation, impacts on soil structure, and crop productivity [99,100] as well as investigations into vertical transport to determine the leaching risk within the soil profile and groundwater [101–104]. Ultimately, identifying the microbial communities responsible for degradation and designing PLA with kinetics explicitly tailored to agricultural crop cycles represent the most promising strategies for aligning material functionality with environmental sustainability [105].

5. Conclusion

PLA remains highly persistent at lower temperatures, whereas degradation and mineralization occur much more rapidly only at elevated temperatures (58 ± 3 °C), resulting in lactic acid production and a decrease in soil pH, with the extent of acidification influenced by the soil buffering capacity. Soil type and amendments (i.e., microcrystalline cellulose) affected the early stages of degradation; however, long-term mineralization was primarily governed by temperature, as demonstrated by the activation energy (E_a), high Q_{10} , and Eyring activation values. Collectively, these findings demonstrate that environmental temperature is the primary determinant of PLA degradation in soils, while soil properties play a secondary, modifying role. These results indicate that PLA is not truly biodegradable in soils at temperatures below its glass transition temperature ($T_g \sim 52$ °C). The application of the ^{14}C -radiolabeling approach was instrumental in establishing these findings by enabling direct quantification of PLA mineralization and tracking of polymer-derived carbon, thereby providing a robust assessment of its environmental fate beyond the capabilities of conventional analytical methods.

CRediT authorship contribution statement

Sara Adeleh: Writing – original draft, Investigation, Data curation.
Tabea Becker: Writing – review & editing.
Sonja Herres-Pawlis: Writing – review & editing, Supervision.
Roland Bol: Writing – review & editing, Supervision.
Birte Drewes: Writing – review & editing.
Thomas Pütz: Writing – review & editing, Supervision.

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.

Acknowledgement

The scientific activities of the Bioeconomy Science Center were financially supported by the Ministry of Culture and Science within the framework of the NRW Strategieprojekt BioSC BioPlastiCycle (No. 313/323–400–002 13). Funding by the Werner Siemens Foundation within the WSS Research Centre "catalaix" is acknowledged. My sincere thanks go to Dr. Lutz Weihermüller (IBG-3: Agrosphere, Forschungszentrum Jülich GmbH) for his invaluable guidance and support with the statistical analysis.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.polyimdeggradstab.2026.112348](https://doi.org/10.1016/j.polyimdeggradstab.2026.112348).

Data availability

Data will be made available on request.

References

- [1] R. Geyer, J.R. Jambeck, K.L. Law, Production, use, and fate of all plastics ever made, *Sci. Adv.* 3 (7) (2017) e1700782, <https://doi.org/10.1126/sciadv.1700782>.
- [2] R. Scalenghe, Resource or waste? A perspective of plastics degradation in soil with a focus on end-of-life options. One step beyond, *Heliyon* 10 (23) (2024) e40647, <https://doi.org/10.1016/j.heliyon.2024.e40647>.
- [3] B. Madrid, S. Wortman, D.G. Hayes, J.M. DeBruyn, C. Miles, M. Flury, T.L. Marsh, S.P. Galinato, K. Englund, S. Agehara, et al., End-of-life management options for agricultural mulch films in the United States—A review, *Front. Sustain. Food Syst.* 2022 6 (2022), <https://doi.org/10.3389/fsufs.2022.921496>. Review.
- [4] M. Sonia Rodríguez-Cruz, M. J. S.M., Pesticides in soils, in: P.G. Jacob de Boer, Gu Ji-Dong, Jones Kevin C, Knepper Thomas P, Negm Abdelazim M (Eds.), *The Handbook of Environmental Chemistry, The Handbook of Environmental Chemistry*, 113, Springer, 2022. Alice Newton, Duc Long Nghiem, Sergi Garcia-Segura.
- [5] Plastics Europe, Bioplastics market development update 2024. <https://www.european-bioplastics.org/market/#>, 2024.
- [6] C. Li, J. Moore-Kucera, J. Lee, A. Corbin, M. Brodhagen, C. Miles, D. Inglis, Effects of biodegradable mulch on soil quality, *Appl. Soil Ecol.* 79 (2014) 59–69, <https://doi.org/10.1016/j.apsoil.2014.02.012>.
- [7] R. Narayan, Misleading claims and misuse proliferate in the nascent, *Bioplast. Mag.* 5 (2010) 38–41.
- [8] ASTM, D883-12 Standard Terminology Relating to Plastics, West Conshohocken, PA, 2012, <https://doi.org/10.1520/D0883-12>.
- [9] S. Teixeira, K.M. Eblagon, F. Miranda, M.F. R. Pereira, J.L. Figueiredo, Towards controlled degradation of poly(lactic) acid in technical applications, *J. Carb. Res.* 7 (2) (2021) 42, <https://doi.org/10.3390/c7020042>.
- [10] N.R. Nair, V.C. Sekhar, K.M. Nampoothiri, A. Pandey, 32 - Biodegradation of biopolymers, in: A. Pandey, S. Negi, C.R. Soccol (Eds.), *Current Developments in Biotechnology and Bioengineering*, Elsevier, 2017, pp. 739–755.
- [11] S. Kubowicz, A.M. Booth, Biodegradability of plastics: challenges and misconceptions, *Environ. Sci. Technol.* ES&T 51 (21) (2017) 12058–12060, <https://doi.org/10.1021/acs.est.7b04051>.
- [12] A.A. Horton, A. Walton, D.J. Spurgeon, E. Lahive, C. Svendsen, Microplastics in freshwater and terrestrial environments: evaluating the current understanding to identify the knowledge gaps and future research priorities, *Sci. Total Environ.* 586 (2017) 127–141, <https://doi.org/10.1016/j.scitotenv.2017.01.190>.
- [13] D. He, Y. Luo, S. Lu, M. Liu, Y. Song, L. Lei, Microplastics in soils: analytical methods, pollution characteristics and ecological risks, *TrAC Trends Anal. Chem.* 109 (2018) 163–172, <https://doi.org/10.1016/j.trac.2018.10.006>.
- [14] E.L. Ng, E. Huerta Lwanga, S.M. Eldridge, P. Johnston, H.W. Hu, V. Geissen, D. Chen, An overview of microplastic and nanoplastic pollution in agroecosystems, *Sci. Total Environ.* 627 (2018) 1377–1388, <https://doi.org/10.1016/j.scitotenv.2018.01.341>.
- [15] A.A. de Souza Machado, C.W. Lau, W. Kloas, J. Bergmann, J.B. Bachelier, E. Faltin, R. Becker, A.S. Görlich, M.C. Rillig, Microplastics can change soil properties and affect plant performance, *Environ. Sci. Technol.* 53 (10) (2019) 6044–6052, <https://doi.org/10.1021/acs.est.9b01339>.
- [16] J. Wang, X. Liu, Y. Li, T. Powell, X. Wang, G. Wang, P. Zhang, Microplastics as contaminants in the soil environment: a mini-review, *Sci. Total Environ.* 691 (2019) 848–857, <https://doi.org/10.1016/j.scitotenv.2019.07.209>.
- [17] Y. Qi, X. Yang, A.M. Pelaez, E. Huerta Lwanga, N. Beriot, H. Gertsens, P. Garbeva, V. Geissen, Macro- and micro- plastics in soil-plant system: effects of plastic mulch film residues on wheat (*Triticum aestivum*) growth, *Sci. Total Environ.* 645 (2018) 1048–1056, <https://doi.org/10.1016/j.scitotenv.2018.07.229>.
- [18] L. Li, Y. Luo, R. Li, Q. Zhou, W.J.G.M. Peijnenburg, N. Yin, J. Yang, C. Tu, Y. Zhang, Effective uptake of submicrometre plastics by crop plants via a crack-entry mode, *Nat. Sustain.* 3 (11) (2020) 929–937, <https://doi.org/10.1038/s41893-020-0567-9>.
- [19] M.A. Abdel-Rahman, Y. Tashiro, K. Sonomoto, Recent advances in lactic acid production by microbial fermentation processes, *Biotechnol. Adv.* 31 (6) (2013) 877–902, <https://doi.org/10.1016/j.biotechadv.2013.04.002>.
- [20] M.A. Abdel-Rahman, Y. Tashiro, K. Sonomoto, Lactic acid production from lignocellulose-derived sugars using lactic acid bacteria: overview and limits, *J. Biotechnol.* 156 (4) (2011) 286–301, <https://doi.org/10.1016/j.jbiotec.2011.06.017>.
- [21] S. Roy, J.W. Rhim, Preparation of bioactive functional poly(lactic acid)/curcumin composite film for food packaging application, *Int. J. Biol. Macromol.* 162 (2020) 1780–1789, <https://doi.org/10.1016/j.jbiomac.2020.08.094>.
- [22] J. Muller, C. González-Martínez, A. Chiralt, Combination of poly(lactic) acid and starch for biodegradable food packaging, *Materials (Basel)* 10 (8) (2017) 952, <https://doi.org/10.3390/ma10080952>.
- [23] P. Baldowska-Witos, K. Piotrowska, W. Kruszelnicka, M. Błaszczak, A. Tomporowski, M. Opielak, R. Kasner, J. Flizikowski, Managing the uncertainty and accuracy of life cycle assessment results for the process of beverage bottle moulding, *Polymers (Basel)* 12 (6) (2020) 1320, <https://doi.org/10.3390/polym12061320>.
- [24] A. Khosravi, A. Fereidoon, M.M. Khorasani, G. Naderi, M.R. Ganjali, P. Zarrintaj, M.R. Saeb, T.J. Gutiérrez, Soft and hard sections from cellulose-reinforced poly(lactic acid)-based food packaging films: a critical review, *Food Packag. Shelf. Life* 23 (2020) 100429, <https://doi.org/10.1016/j.fpsl.2019.100429>.
- [25] I. Gan, W.S. Chow, Antimicrobial poly(lactic acid)/cellulose bionanocomposite for food packaging application: a review, *Food Packag. Shelf. Life* 17 (2018) 150–161, <https://doi.org/10.1016/j.fpsl.2018.06.012>.
- [26] T.P. Haider, C. Völker, J. Kramm, K. Landfester, F.R. Wurm, Plastics of the future? The impact of biodegradable polymers on the environment and on society, *Angew. Chem. Int. Ed.* 58 (1) (2019) 50–62, <https://doi.org/10.1002/anie.201805766>.
- [27] K.L.G. Ho, A.L. Pometto, P.N. Hinz, A. Gadea-Rivas, J.A. Briceño, A. Rojas, Field exposure study of polylactic acid (PLA) plastic films in the banana fields of Costa Rica, *J. Environ. Polym. Degrad.* 7 (4) (1999) 167–172, <https://doi.org/10.1023/A:1022849813748>.
- [28] E. Rudnik, D. Briassoulis, Degradation behaviour of poly(lactic acid) films and fibres in soil under Mediterranean field conditions and laboratory simulations testing, *Ind. Crops. Prod.* 33 (3) (2011) 648–658, <https://doi.org/10.1016/j.indcrop.2010.12.031>.

- [29] R. Auras, B. Harte, S. Selke, An overview of polylactides as packaging materials, *Macromol. Biosci.* 4 (9) (2004) 835–864, <https://doi.org/10.1002/mabi.200400043>.
- [30] R.H. Platel, L.M. Hodgson, C.K. Williams, Biocompatible initiators for lactide polymerization, *Polym. Rev.* 48 (1) (2008) 11–63, <https://doi.org/10.1080/15583720701834166>.
- [31] S. Momeni, K. Craplewe, M. Sadfer, S. Luz, D. Sauvageau, A. Elias, Accelerating the biodegradation of poly(lactic acid) through the inclusion of plant fibers: a review of recent advances, *ACS Sustain. Chem. Eng.* 11 (42) (2023) 15146–15170, <https://doi.org/10.1021/acsschemeng.3c04240>.
- [32] N. Hajilou, S.S. Mostafay, A.L. Yarin, T. Shokuhfar, A comparative review on biodegradation of poly(Lactic Acid) in soil, compost, water, and wastewater environments: incorporating mathematical modeling perspectives, *Appl. Chem.* 5 (1) (2025) 1, <https://doi.org/10.3390/appliedchem5010001>.
- [33] S. Liu, B. Chen, K. Wang, J. Wang, K. Wang, Y. Suo, X. Yang, Y. Zhu, J. Zhang, M. Lu, et al., Unveiling the impact of biodegradable polylactic acid microplastics on meadow soil health, *Environ. Geochem. Health* 47 (2) (2025) 45, <https://doi.org/10.1007/s10653-025-02358-3>.
- [34] S. Lai, C. Fan, P. Yang, Y. Fang, L. Zhang, M. Jian, G. Dai, J. Liu, H. Yang, L. Shen, Effects of different microplastics on the physicochemical properties and microbial diversity of rice rhizosphere soil, *Front. Microbiol.* 2025 15 (2024), <https://doi.org/10.3389/fmicb.2024.1513890>. Original Research.
- [35] F. Guo, B. Liu, J. Zhao, Y. Hou, J. Wu, C. Zhou, H. Hu, T. Zhang, Z. Yang, Effects of polyethylene, polylactic acid, and tire particles on the sediment microbiome and metabolome at high and low temperatures, *Appl. Environ. Microbiol.* 90 (2) (2024) 90, <https://doi.org/10.1128/aem.02016-23>, e02016-02023.
- [36] S. Mariano, S. T. M. Fidaleo, M. Rossi, L. Dini, Micro and nanoplastics identification: classic methods and innovative detection techniques, *Front. Toxicol.* 3 (2021), <https://doi.org/10.3389/ftox.2021.636640>.
- [37] C. Junhao, Z. Xining, G. Xiaodong, Z. Li, H. Qi, K.H.M. Siddique, Extraction and identification methods of microplastics and nanoplastics in agricultural soil: a review, *J. Environ. Manage.* 294 (2021) 112997, <https://doi.org/10.1016/j.jenvman.2021.112997>.
- [38] C.N. Perez, F. Carré, A. Hoarou-Belkhir, A. Joris, P.E.G. Leonards, M.H. Lamoree, Innovations in analytical methods to assess the occurrence of microplastics in soil, *J. Environ. Chem. Eng.* 10 (3) (2022) 107421, <https://doi.org/10.1016/j.jece.2022.107421>.
- [39] R.J. Diamond, *Labelled Polymers - Synthesis, Analysis and Degradation Studies*, University of Surrey, 1988.
- [40] S. Ponsart, J. Coudane, J.L. Morgat, M. Vert, Synthesis of 3H and fluorescence-labelled poly (DL-Lactic acid), *J. Label. Compd. Radiopharm.* 44 (10) (2001) 677–687, <https://doi.org/10.1002/jlcr.495>.
- [41] M. Karamanlioglu, G.D. Robson, The influence of biotic and abiotic factors on the rate of degradation of poly(lactic) acid (PLA) coupons buried in compost and soil, *Polym. Degrad. Stab.* 98 (10) (2013) 2063–2071, <https://doi.org/10.1016/j.polymdegradstab.2013.07.004>.
- [42] S. Lv, Y. Zhang, J. Gu, H. Tan, Biodegradation behavior and modelling of soil burial effect on degradation rate of PLA blended with starch and wood flour, *Colloids Surf. B* 159 (2017) 800–808, <https://doi.org/10.1016/j.colsurf.2017.08.056>.
- [43] K.L.G. Ho, A.L. Pometto, A. Gadea-Rivas, J.A. Briceño, A. Rojas, Degradation of polylactic acid (PLA) plastic in Costa Rican soil and Iowa State University compost rows, *J. Environ. Polym. Degrad.* 7 (4) (1999) 173–177, <https://doi.org/10.1023/A:1022874530586>.
- [44] S. Lv, X. Liu, J. Gu, Y. Jiang, H. Tan, Y. Zhang, Microstructure analysis of polylactic acid-based composites during degradation in soil, *Int. Biodeterior. Biodegrad.* 122 (2017) 53–60, <https://doi.org/10.1016/j.ibiod.2017.04.017>.
- [45] Chuensangjun, C.; Pechyen, C.; Sirisansaneeyakul, S. Degradation behaviors of different blends of polylactic acid buried in soil. *Energy procedia* 2013, 34, 73–82, DOI: <https://doi.org/10.1016/j.egypro.2013.06.735>.
- [46] G. Rajesh, A.V. Ratna Prasad, A.V.S.K.S. Gupta, Soil degradation characteristics of short sisal/PLA composites, *Mater. Tod. Proc.* 18 (2019) 1–7, <https://doi.org/10.1016/j.matpr.2019.06.270>.
- [47] K. Janczak, K. Hryniewicz, Z. Znajewska, G. Dąbrowska, Use of rhizosphere microorganisms in the biodegradation of PLA and PET polymers in compost soil, *Int. Biodeterior. Biodegrad.* 130 (2018) 65–75, <https://doi.org/10.1016/j.ibiod.2018.03.017>.
- [48] A. Torres, S.M. Li, S. Roussos, M. Vert, Poly(lactic acid) degradation in soil or under controlled conditions, *J. Appl. Polym. Sci.* 62 (13) (1996) 2295–2302, [https://doi.org/10.1002/\(SICI\)1097-4628\(19961226\)62:13<2295::AID-PP14>3.0.CO;2-2](https://doi.org/10.1002/(SICI)1097-4628(19961226)62:13<2295::AID-PP14>3.0.CO;2-2).
- [49] L. Santonja-Blasco, R. Moriana, J.D. Badía, Ribes-Greus, A. Thermal analysis applied to the characterization of degradation in soil of polylactide: I. Calorimetric and viscoelastic analyses, *Polym. Degrad. Stab.* 95 (11) (2010) 2185–2191, <https://doi.org/10.1016/j.polymdegradstab.2010.08.005>.
- [50] Y. Wu, Z. Wang, Y. Yu, X. Liu, D. Wang, J. Wang, Y. Li, B. Zhu, J.K. Whalen, H. Yao, Degrading poly(lactic acid) microplastic induces priming in agricultural soils, *Appl. Soil Ecol.* 206 (2025) 105911, <https://doi.org/10.1016/j.apsoil.2025.105911>.
- [51] S. Adeleh, T. Becker, S. Herres-Pawlitz, B. Bol, B. Drewes, T. Pütz, Synthesis of micro 14C-labeled polylactide for environmental assessment analysis, *Chem. Meth.* 5 (2025) e202500087, <https://doi.org/10.1002/cmt.202500087>.
- [52] A.S. Karlsson, L. Weihermüller, W. Tappe, S. Mukherjee, S. Spielvogel, Field scale boscalid residues and dissipation half-life estimation in a sandy soil, *Chemosphere* 145 (2016) 163–173, <https://doi.org/10.1016/j.chemosphere.2015.11.026>.
- [53] J. Koestel, R. Kasteel, A. Kemna, O. Esser, M. Javaux, A. Binley, H. Vereecken, Imaging brilliant blue stained soil by means of electrical resistivity tomography, *Vadose Zone J.* 8 (4) (2009) 963–975.
- [54] S. Garré, M. Javaux, J. Vanderborght, L. Pagès, H. Vereecken, Three-dimensional electrical resistivity tomography to monitor root zone water dynamics, *Vadose Zone J.* 10 (1) (2011) 412–424.
- [55] K.K. Thomas Pütz, *Profilbeschreibung Und Analysenergebnisse des Standortes Kaldenkirchen - Hülst; IRA 6/91, Institut für Radioagronomie - Forschungszentrum Jülich, Institut für Bodenkunde - Rheinische-Friedrich-Wilhelms-Universität Bonn*, 1991.
- [56] M. Burkhardt, *Profilbeschreibung Und Analysenergebnisse für den Standort Merzenhausen; IRA 1/99, Institut für Radioagronomie - Forschungszentrum Jülich, Institut für Bodenkunde - Rheinische-Friedrich-Wilhelms-Universität Bonn*, 1999.
- [57] S. Pätzold, *Profilbeschreibung Merzenhausen Institut für Bodenkunde* (2001).
- [58] J. Forster, *Soil sampling, handling, storage and analysis. Methods in Applied Soil Microbiology and Biochemistry*, Elsevier, 1995, pp. 49–121.
- [59] W.A. Jury, R. Horton, *Soil Physics*, Wiley, 2004.
- [60] ASTM. Standard test method for determining aerobic biodegradation of plastic materials in soil-D5988–18; 2018. DOI: [10.1520/D5988-18](https://doi.org/10.1520/D5988-18).
- [61] ISO. *Plastics — Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved-ISO 17556:2019*; 2019.
- [62] D.C. Coleman, *Carbon Isotope Techniques*, Academic Press, 2012.
- [63] A.B. Gillmore, C.A. Steren, D.G. Hayes, S.M. Schaeffer, Quantification of polybutylene adipate terephthalate-based micro- and nano-plastics from soil using proton nuclear magnetic resonance spectroscopy, *JoVE* (220) (2025) e67471, <https://doi.org/10.3791/67471>.
- [64] D. Hennecke, A. Bauer, M. Herrchen, E. Wischerhoff, F. Gores, Cationic polyacrylamide copolymers (PAMs): environmental half life determination in sludge-treated soil, *Environ. Sci. Eur.* 30 (1) (2018) 16, <https://doi.org/10.1186/s12302-018-0143-3>.
- [65] FOCUS. Guidance Document on Estimating Persistence and Degradation Kinetics from Environmental Fate Studies on Pesticides in EU Registration; Brussels, Belgium, 2006. https://esdac.jrc.ec.europa.eu/public_path/projects_data/focus/dk/docs/finalreportFOCDegKinetics.pdf.
- [66] FOCUS. Generic Guidance for Estimating Persistence and Degradation Kinetics from Environmental Fate Studies on Pesticides in EU Registration; Brussels, Belgium, 2014. https://esdac.jrc.ec.europa.eu/public_path/projects_data/focus/dk/docs/FOCUSkineticsv1.1Dec2014.pdf.
- [67] A. Abubakar, M.B.H.A. Rahim, M.E. Khayat, Activation energy, temperature coefficient and Q10 value estimations of the growth of *Rhodotorula* sp. Strain MBH23 on Acrylamide, *J. Environ. Microbiol. Toxicol.* 12 (1) (2024) 1–6.
- [68] P. Straka, Activation entropy of degradation of cross-linked polymer in catalyzed and uncatalyzed slow pyrolysis, *Next Materials* 11 (2026) 101746, <https://doi.org/10.1016/j.nxmate.2026.101746>.
- [69] V.V. Krongauz, Compensation effect: sublimation, diffusion in polymers, polymer degradation, *J. Therm. Anal. Calorim.* 138 (5) (2019) 3425–3444, <https://doi.org/10.1007/s10973-019-08851-z>.
- [70] S.D. Stephan Mantel, *Sepp Deckers, Word Reference Base for Soil Resources*, Elsevier, 2023.
- [71] P. Schad, *World Reference Base for Soil Resources—Its fourth edition and its history*, *J. Plant Nutr. Soil Sci.* 186 (2) (2023) 151–163, <https://doi.org/10.1002/jpln.202200417>.
- [72] N.A. Rosli, M. Karamanlioglu, H. Kargazadeh, I. Ahmad, Comprehensive exploration of natural degradation of poly(lactic acid) blends in various degradation media: a review, *Int. J. Biol. Macromol.* 187 (2021) 732–741, <https://doi.org/10.1016/j.ijbiomac.2021.07.196>.
- [73] H. Yagi, F. Ninomiya, M. Funabashi, M. Kunioka, Anaerobic biodegradation tests of poly(lactic acid) under mesophilic and thermophilic conditions using a new evaluation system for methane fermentation in Anaerobic sludge, *Int. J. Mol. Sci.* 10 (9) (2009) 3824–3835.
- [74] E.D. Vance, P.C. Brookes, D.S. Jenkinson, An extraction method for measuring soil microbial biomass C, *Soil. Biol. Biochem.* 19 (6) (1987) 703–707, [https://doi.org/10.1016/0038-0717\(87\)90052-6](https://doi.org/10.1016/0038-0717(87)90052-6).
- [75] C. Vasile, D. Pamfil, M. Răpă, R.N. Darie-Niță, A.C. Mitelut, E.E. Popa, P. A. Popescu, M.C. Draghici, M.E. Popa, Study of the soil burial degradation of some PLA/CS biocomposites, *Compos. Part B* 142 (2018) 251–262, <https://doi.org/10.1016/j.compositesb.2018.01.026>.
- [76] E. Rudnik, D. Briassoulis, Comparative biodegradation in soil behaviour of two biodegradable polymers based on renewable resources, *J. Polym. Environ.* 19 (1) (2011) 18–39, <https://doi.org/10.1007/s10924-010-0243-7>.
- [77] H.P. Blume, G.W. Brümmer, R. Horn, E. Kandeler, I. Kögel-Knabner, R. Kretzschmar, K. Stahr, B.M. Wilke, S. Thiele-Bruhn, G. Welp, Scheffer/Schachtschabel *Soil Science*, Springer, 2016, <https://doi.org/10.1007/978-3-642-30942-7>.
- [78] S. Muniyasamy, O. Ofosu, M.J. John, R.D. Anandjiwala, Mineralization of poly(lactic acid) (PLA), poly(3-hydroxybutyrate-co-valerate) (PHBV) and PLA/PHBV blend in compost and soil environments, *J. Renew. Mater.* 4 (2) (2016) 133–145, <https://doi.org/10.7559/JRM.2016.634104>.
- [79] M. Karamanlioglu, A. Houlden, G.D. Robson, Isolation and characterisation of fungal communities associated with degradation and growth on the surface of poly(lactic acid) (PLA) in soil and compost, *Int. Biodeterior. Biodegrad.* 95 (2014) 301–310, <https://doi.org/10.1016/j.ibiod.2014.09.006>.
- [80] S.M. Satti, A.A. Shah, T.L. Marsh, R. Auras, Biodegradation of poly(lactic acid) in soil microcosms at ambient temperature: evaluation of natural attenuation, bio-

- augmentation and bio-stimulation, *J. Polym. Environ.* 26 (9) (2018) 3848–3857, <https://doi.org/10.1007/s10924-018-1264-x>.
- [81] J.J. Kolstad, E.T.H. Vink, B. De Wilde, L. Debeer, Assessment of anaerobic degradation of Ingeo™ polylactides under accelerated landfill conditions, *Polym. Degrad. Stab.* 97 (7) (2012) 1131–1141, <https://doi.org/10.1016/j.polymdegradstab.2012.04.003>.
- [82] G. Kale, R. Auras, S.P. Singh, R. Narayan, Biodegradability of polylactide bottles in real and simulated composting conditions, *Polym. Test.* 26 (8) (2007) 1049–1061, <https://doi.org/10.1016/j.polymertesting.2007.07.006>.
- [83] C. Boonmee, C. Kositanont, T. Leejarkpai, Degradation of poly (lactic acid) under simulated landfill conditions. *Environment and, Nat. Resour. J.* 14 (2) (2016) 1–9, <https://doi.org/10.14456/enrj.2016.8>.
- [84] R. Ray, N.C.B. Weil, *The Nature and Properties of Soils*, Pearson, 2017.
- [85] G. Sposito, *The Chemistry of Soils*, Oxford university press, 2008.
- [86] X. Ling, Y. Gao, R. Li, R. Chang, Y. Li, W. Xiao, A cost-effective standardized quantitative detection method for soil microplastics in different substrates, *Toxics* 14 (105) (2026) 105, <https://doi.org/10.3390/toxics14010105>.
- [87] D. Thomas, B. Schütze, W.M. Heinze, Z. Steinmetz, Sample preparation techniques for the analysis of microplastics in soil—A review, *Sustainability* 12 (9074) (2020) 9074, <https://doi.org/10.3390/su12219074>.
- [88] S. Peneva, Q.N.P. Le, D.R. Munhoz, O. Wrigley, F. Wille, H. Doose, C. Halsall, P. Harkes, M. Sander, M. Braun, et al., Microplastic analysis in soils: a comparative assessment, *Ecotoxicol. Environ. Saf.* 289 (2025) 117428, <https://doi.org/10.1016/j.ecoenv.2024.117428>.
- [89] S. Yousafzai, M. Farid, M. Zubair, N. Naeem, W. Zafar, Z.U. Zaman Asam, S. Farid, S. Ali, Detection and degradation of microplastics in the environment: a review, *Environ. Sci. Adv.* 4 (8) (2025) 1142–1165, <https://doi.org/10.1039/D5VA00064E>, 10.1039/D5VA00064E.
- [90] F. Ruggero, E. Carretti, R. Gori, T. Lotti, C. Lubello, Monitoring of degradation of starch-based biopolymer film under different composting conditions, using TGA, FTIR and SEM analysis, *Chemosphere* 246 (2020) 125770, <https://doi.org/10.1016/j.chemosphere.2019.125770>.
- [91] S. Baidurah, Methods of analyses for biodegradable polymers: a review, *Polymers (Basel)* 14 (4928) (2022) 4928, <https://doi.org/10.3390/polym14224928>.
- [92] L. Jeszeová, A. Puškárová, M. Bučková, L. Kraková, T. Grivalský, M. Danko, K. Mosnáčková, Š. Chmela, D. Pangallo, Microbial communities responsible for the degradation of poly(lactic acid)/poly(3-hydroxybutyrate) blend mulches in soil burial respirometric tests, *World J. Microbiol. Biotechnol.* 34 (7) (2018) 101, <https://doi.org/10.1007/s11274-018-2483-y>.
- [93] Y. Zhang, W. Gao, A. Mo, J. Jiang, D. He, Degradation of polylactic acid/polybutylene adipate films in different ratios and the response of bacterial community in soil environments, *Environ. Pollut.* 313 (2022) 120167, <https://doi.org/10.1016/j.envpol.2022.120167>.
- [94] M. Zhang, H. Jia, Y. Weng, C. Li, Biodegradable PLA/PBAT mulch on microbial community structure in different soils, *Int. Biodeterior. Biodegrad.* 145 (2019) 104817, <https://doi.org/10.1016/j.ibiod.2019.104817>.
- [95] Z. Saadi, A. Rasmont, G. Cesar, H. Bewa, L. Benguigui, Fungal degradation of poly (l-lactide) in soil and in compost, *J. Polym. Environ.* 20 (2) (2012) 273–282, <https://doi.org/10.1007/s10924-011-0399-9>.
- [96] M.Y. Kim, C. Kim, J. Moon, J. Heo, S.P. Jung, J.R. Kim, Polymer film-based screening and isolation of polylactic acid (PLA)-degrading microorganisms, *J. Microbiol. Biotechnol.* 27 (2) (2017) 342–349.
- [97] S. Lambert, *Environmental Risk of Polymer and Their Degradation Products*, University of York, 2013.
- [98] C. Wang, K. Zhao, Y. Li, S. Hu, A life-cycle based integrated framework for assessing the sustainability of plastic additives, *ACS Sustain. Chem. Eng.* 13 (33) (2025) 13399–13413, <https://doi.org/10.1021/acssuschemeng.5c04469>.
- [99] M. Gencer, M.O. Akca, M. Acar, Z.S. Abdelrazik, T.S. Wahab, S. Gündoğdu, O. C. Turgay, Microplastic accumulation and soil quality changes under varying plastic mulching durations in the Mediterranean region, *J. Soil. Sci. Plant Nutr.* 26 (1) (2026) 2513–2528, <https://doi.org/10.1007/s42729-026-03010-4>.
- [100] I. de Sadeleer, A. Woodhouse, Environmental impact of biodegradable and non-biodegradable agricultural mulch film: a case study for Nordic conditions, *Int. J. Life Cycle Assess.* 29 (2) (2024) 275–290, <https://doi.org/10.1007/s11367-023-02253-y>.
- [101] A. Maqbool, W. Li, C. Stumpp, M.A. Soriano, J.A. Gómez, Effect of preferential microplastics leaching through macropores on vertical soil particle transport, *Eur. J. Soil. Sci.* 76 (3) (2025) e70140, <https://doi.org/10.1111/ejss.70140>.
- [102] Y. Qiu, S. Zhou, C. Zhang, L. Chen, W. Qin, Q. Zhang, Vertical distribution and weathering characteristic of microplastics in soil profile of different land use types, *Sci. Total. Environ.* 905 (2023) 166902, <https://doi.org/10.1016/j.scitotenv.2023.166902>.
- [103] H. Luo, L. Chang, T. Ju, Y. Li, Factors influencing the vertical migration of microplastics up and down the soil profile, *ACS Omega* 9 (51) (2024) 50064–50077, <https://doi.org/10.1021/acsomega.4c04083>.
- [104] A. Kumari, S. Chakraborty, Vertical profiling of micro-plastics in agricultural soils: polymer types, metal interactions, and implication on crop rooting zones, *Environ. Monit. Assess.* 198 (4) (2026) 291, <https://doi.org/10.1007/s10661-026-15148-w>.
- [105] M.P. Ginige, A.C. Warden, A.H. Kaksonen, Towards a circular plastics economy: synchronising material design, hybrid processing, digital logistics, and adaptive policy, *Rev. Environ. Sci. Bio/Tech.* 25 (2) (2026) 22, <https://doi.org/10.1007/s11157-026-09773-7>.