Detectability of degradable organic matter in agricultural soils by thermogravimetry

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Abstract

Sustainable agricultural land use requires an assessment of degradable soil organic matter (SOM) because of its key function for soil fertility and plant nutrition. Such an assessment for practical land use should consider transformation processes of SOM and its sources of different origin. In this study, we combined a 120-day incubation experiment with thermal decay dynamics of agricultural soils altered by added organic amendments. The aim was to determine the abilities and limits of thermal analysis as a rapid approach revealing differences in the degradability of SOM. The carried out experiments based on two independent sampling sets. The first sample set consisted of soil samples taken from non-fertilized plots of three German long-term agricultural field experiments (LTAEs), then artificially mixed with straw, farmyard manure, sheep faeces, and charcoal equal to 60 Mg ha^{-1} under laboratory conditions. The second sample set based on soil samples of different treatments (*e.g.*, crop type, fertilization, cultivation) in LTAEs at Bad Lauchstädt and Müncheberg, Germany. Before and after the incubation experiment, thermal mass losses (TML) at selected temperatures were determined by thermogravimetry indicating the degradability of organic amendments mixed in soils. The results confirmed different microbial degradability of organic amendments and SOM under laboratory conditions. Thermal decay dynamics revealed incubation-induced changes in the artificial soil mixtures primarily at TML around 300°C in the case of applied straw and sheep faeces, whereas farmyard manure showed mainly changes in TML around 450°C. Charcoal did not show significant degradation during incubation, which was confirmed by TML. Detailed analyses of the artificial soil mixtures revealed close correlations between CO_2-C evolution during incubation and changes in TML at 300°C with $R^2 > 0.96$. Results of the soils from LTAEs showed similar incubation-induced changes in thermal decay dynamics for fresh plant residues and farmyard manure. We conclude that the practical assessment of SOM could be facilitated by thermal decay dynamics if modified sample preparation and evaluation algorithms are used beyond traditional peak analysis.

Key words: artificial soil mixtures / field experiments / microbial degradation / organic fertilizers / soil organic matter / thermal decay dynamics

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1 Introduction

Soils consist of different biological, organic and mineral components (*Totsche* et al., 2010) and support the primary production of food, fibers, animal feed, and fuel (*Weil* and *Brady*, 2016). Soil organic matter (SOM) is commonly recognized as the most crucial component of soils determining its physical, chemical and biological properties and ecosystem productivity (*Gregorich* et al., 2015). SOM is of great importance for the global carbon cycle as a very large store of terrestrial carbon, which is vulnerable to climate change (*Field* et al., 2007; *Conant* et al., 2011). The permanent transformation and min-

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eralization of SOM is dependent on temperature, moisture, soil texture, its composition and other factors (*von Lützow* et al., 2006; *Shen* et al., 2009). The primary sources of SOM are decomposed plant residues (*Weil* and *Brady*, 2016), while in agricultural soils, organic fertilizers are also a major component (*e.g*., straw, farmyard manure and charcoal). Organic fertilizers are frequently applied to increase the SOM content, improve biological activity, nutrient availability, soil structure, chemical properties, and crop yields (*Haynes* and *Naidu*, 1998; *Bending* and *Turner*, 1999; *Körschens* et al., 2013; *Schröder* et al., 2016).

The biological transformation of organic amendments applied to soils is regulated by their chemical composition (*Johnson* et al., 2007), physical structure (*Prescott*, 2010), soil texture and quantity as well. They can consist of easily biodegradable or stabilized organic carbon or both (*Sarma* et al., 2017). For example, straw as a biodegradable organic amendment is decomposed relatively rapidly by microorganisms which is reflected in high degradation rates (*Powlson* et al., 2011; *Hansen* et al., 2016). In contrast, farmyard manure contains both biodegradable and stabilized organic carbon and has longer turnover rates in soil environments (*Körschens* et al., 1994; *Haynes* and *Naidu*, 1998; *Li* et al., 2018a). Another frequently used fertilizer is charcoal, which is characterized by its superior stability in soils compared to other organic amendments (*Glaser* et al., 2002; *Kuzyakov* et al., 2009; *Li* et al., 2018b). The assessment of such organic amendments in SOM found increasing attention in the last years, because of their influence on soil organic carbon (SOC) stability and biological transformation processes in soil environments (*Mohanty* et al., 2011; *Plante* et al., 2011; *Kučerík* et al., 2018). The complexity of SOM which results from different sources (*Ferreras* et al., 2006; *Kätterer* et al., 2014), different transformation mechanisms and environmental controls of SOM stabilization (*Franko* and *Merbach*, 2017) makes the development of methods for assessing biological degradability a challenging task. Recently, artificial mixing of soils with organic substances under laboratory conditions (model experiments) were proposed to substitute previous attempts to develop quality indicators and recovery rates for SOM (*Fernández-Gálvez* et al., 2012; *Jin* et al., 2016; *Nguyen* et al., 2016; *Tokarski* et al., 2018). However, they are characterized by limited applicability to agricultural practice. Other authors prefer long-term agricultural field experiments (LTAEs) to investigate the long-term impact of SOM on plant nutrition, crop yield, and climate change and to focus on practical issues (*Körschens* et al., 2013; *Dignac* et al., 2017). However, these experiments are very expensive, long-lived and may not provide necessary answers to farmer's current questions about the assessment of short-term changes of SOM content, fertilizers and soil management strategies. Therefore, the assessment of the biodegradability of SOM remains an unsolved task (*Sánchez* et al., 2008).

Two simple analytical approaches and their interrelationship have been thoroughly discussed in the past: (1) soil respiration as an indicator of microbial stability (*Plante* et al., 2011; *Siewert* et al., 2012; *Peltre* et al., 2013; *Pronk* et al., 2013) and (2) thermal analysis techniques (*e.g*., thermogravimetry, differential thermal analysis and differential scanning calorimetry) for rapid determination of soil properties and SOM stability (*Siewert*, 2004; *Lopez-Capel* et al., 2005; *Plante* et al., 2009; *Gregorich* et al., 2015; *Siewert* and *Kučerík*, 2015; *Kučerík* et al., 2016; *Kristl* et al., 2016; *Tokarski* et al., 2018; *Kučerík* et al., 2018; *Soucémarianadin* et al., 2018).

Measuring microbial respiration by degradation of SOM during incubation experiments offers a valuable but very time consuming way to characterize SOM (*Paul* et al., 2006; *Mohanty* et al., 2011; *Peltre* et al., 2013; *Nguyen* et al., 2016; *Schiedung* et al., 2016; *Xu* et al., 2017) and its interactions with minerals (*Pronk* et al., 2013). This method provides information about both stabilized and biodegradable fraction of soil organic carbon that is available in soils to heterotrophic organisms (*Hopkins*, 2007). The long-lasting and expensive experimental procedure, however, limit its application for agriculture practice.

Thermal analysis was used to investigate the biological stability of SOM, but provided contradictory results in the assessment of SOC transformation processes (*Otero* et al., 2002; *Peltre* et al., 2013; *Kučerík* and *Siewert*, 2014; *Barros* et al., 2016; *Schiedung* et al., 2017). Nevertheless, the consideration of natural soils not influenced by human activities and gentle soil sample preparation with standardized water content opened new opportunities, *e.g*., for soil property prediction (organic carbon, nitrogen, and clay content) and for monitoring microbial degradation of SOM (*Siewert*, 2001; *Siewert*, 2004; *Siewert* et al., 2012; *Kučerík* et al., 2016). *Tokarski* et al. (2018) detected SOM components in soils independent of clay content using thermal decay dynamics recorded by thermogravimetry. Several authors recommend a consideration of the nature and thermal stability of SOM associated with clay (*Plante* et al., 2005; *Hassink*, 1997) because it might affect its microbial degradability. However, these findings could not answer questions to what extent the degradability of SOM and its components such as organic amendments can be determined considering the content of SOC, clay and factors that regulate SOM accumulation.

This study focuses on the detectability of changes in thermal decay dynamics caused by differences in microbial degradation of SOM in laboratory incubation experiments. The aim of this study was to discover and validate temperature ranges with specific characteristics of the microbial degradability of SOM and organic fertilizers in soils with different clay contents using dynamics of thermal mass losses.

2 Material and methods

The thermal decay dynamics of soils from two independent sample sets were recorded before and after 120 days of incubation under laboratory conditions. First sample set based on three soils from non-fertilized plots of different long-term agricultural field experiments (LTAEs) which were spiked with different organic amendments under laboratory conditions. Second sample set focuses on soil samples from LTAEs with different organic long-term fertilization and cultivation techniques.

2.1 Description of the used soil samples and experimental design

2.1.1 Characteristics of experimental sites

For the study soils were used from three different LTAEs in Germany. The first site is located at the Helmholtz Centre for Environmental Research (UFZ) in Bad Lauchstädt (Saxony-Anhalt, 51°24′N, 11°53′E) with a mean annual precipitation of 481 mm and a mean temperature of 8.9°C on a silty loam soil developed in loess with a clay content of around 210 g kg^{-1} soil (*Franko* and *Merbach*, 2017). The soil is classified as a Haplic Chernozem (WRB). The second site is situated at the Leibniz Institute of Vegetable and Ornamental Crops (IGZ) in Grossbeeren (52°21′N, 13°19′E, Brandenburg) with a mean annual precipitation of 521 mm and a mean temperature of 8.4°C (*Rühlmann*, 2009). The soil is a silty sand with a clay content of around 55 g kg^{-1} soil and classified as an Arenic-Luvisol (WRB). The third site belongs to the Leibniz Centre for Agricultural Landscape Research (ZALF) in Müncheberg (52°30′N, 14°6′E, Brandenburg) and had a mean annual precipitation of 525 mm and a mean temperature of 8.6°C (*Barkusky*, 2009). The soil is a loamy sand with a clay content of around 50 g kg^{-1} soil and classified as an Albic Luvisol (WRB).

2.1.2 Soil samples

Artificial mixtures with organic amendments under laboratory conditions

For the investigation of artificial mixtures of soils with organic amendments, samples of non-fertilized plots of LTAEs in Bad Lauchstädt (Static Fertilization Experiment, established 1902, 16 g SOC kg^{-1}), Grossbeeren (Static Fertilization Experiment, established 1989, 8 g SOC kg^{-1} soil), and Müncheberg (Nutrition Increase Experiment, established 1963, 5 g SOC kg^{-1} soil) were used. Detailed description of the LTAEs were given by *Barkusky* (2009), *Rühlmann* (2009), and *Körschens* and *Pfefferkorn* (1998). These soil samples were mixed with straw (ST), farmyard manure (FYM), sheep faeces (SF), and charcoal (CC) under laboratory conditions (see details in *Tokarski* et al., 2018). Each soil was spiked with an equivalent of 60 Mg ha^{-1} of fresh matter (1.3–7.5 g C kg^{-1} soil) of all four amendments (n = 12, see Tab. 1). The added quantity of organic amendments represents the highest fertilization rates in these LTAEs (*Körschens* et al., 2013).

Long-term agricultural field experiments (LTAEs)

This sample set consisted of soils from different treatments of LTAEs in Bad Lauchstädt and Müncheberg with different long-term organic fertilization and cultivation treatments.

From Bad Lauchstädt we used soil samples of two LTAEs. One LTAE was started in 1983 as Incremental Manure Experiment (V494) to study the impact of high quantities of farmyard manure on yield and soil properties. It includes four different quantities of farmyard manure: 0, 50, 100, and 200 Mg ha^{-1} y^{-1} (factor A, four levels) applied to corn and bare fallow (factor B, two crop rotations) with two field repetitions (8 treatments, 16 plots, details see in *Körschens* and *Pfefferkorn*, 1998). Samples were taken from bare fallow and corn treatments without application of farmyard manure (non-fertilized control, 20–23 g SOC kg^{-1} soil) and with highest application rate of FYM (200 Mg farmyard manure ha^{-1} y^{-1} or 40–46 g SOC kg^{-1} soil). In total, we analyzed eight samples from this LTAE (2 fertilizer levels × 2 crop rotations × 2 field repetitions).

The second LTAE was established in 1988 as the Fallow Experiment (V505a) with four field repetitions in order to investigate the impact of (1) mechanical tillage (mechanical bare fallow), (2) herbicide application (chemical bare fallow), (3) combination of mechanical and herbicide bare fallow, and (4) succession of weed flora (self-greening) (*Franko* and *Merbach*, 2017) on soil properties. The sampling for this study was carried out on treatments with mechanical bare fallow (18 g SOC kg^{-1} soil) and with succession of weed flora (self-greening) (27 g SOC kg^{-1} soil) only. A total of eight samples was collected and investigated (2 treatments × 4 field replications).

The LTAE in Müncheberg is designated as V140 and focuses on the influence of different mineral and organic fertilizers on yield and soil fertility. It was established in a full randomized block design in 1963. The LTAE consist of 8 field repetitions for treatments with different mineral nitrogen levels (40 to 200 kg ha^{-1} y^{-1}), organic fertilization with farmyard manure (4.8–12.8 Mg ha^{-1} dry matter every third year) and with straw (4.0 Mg ha^{-1} dry matter every second year) (*Barkusky*, 2009). The sample set consisted of four repetitions and included treatments with non-fertilization (6 g SOC kg^{-1} soil), with mineral nitrogen fertilization (6 g SOC kg^{-1} soil), with high application of farmyard manure (7 g SOC kg^{-1} soil), and with straw application (7 g SOC kg^{-1} soil) (4 plots × 4 replications = 16 samples).

Table 1: Basic properties of added straw (ST), farmyard manure (FYM), sheep faces (SF), and charcoal (CC) and expected changes in carbon (C) content by adding 60 Mg ha^{-1} fresh matter of organic amendments.

Type of organic amendments		**ST**	**FYM**	**SF**	**CC**
Dry matter content (%)		86	23	35	80
carbon content in dry mass (g kg^{-1})		402	413	357	706
Added quantity of organic amendments (fresh matter)	**Mg ha^{-1} OA[a]**	**predicted increase in carbon content (g C kg^{-1} soil)**			
	60	+4.6	+1.3	+1.7	+7.5

[a]OA: Organic amendments.

Samples were taken in the second year after straw incorporation, in the first year after application of farmyard manure and after yielding potatoes.

2.2 Sampling and sample preparation

All soil samples were taken in autumn 2015 from the selected treatments as a mixed sample (≈ 30 cores were mixed to gain one composite sample) at a depth of 0–25 cm. The soil samples were immediately gently air dried and sieved to pass a 2-mm screen. In order to create the same conditions of moisture equilibrations (comparable contents of bound water), the subsequent sample preparation included storing at 76% relative humidity for at least two weeks (at 22°C) in desiccators above saturated NaCl (see details in *Siewert*, 2001).

For the investigations of the artificial soil mixtures, the organic amendments were ground to 0.2 mm particle size (Retsch Ultra Centrifugal Mill ZM 200) after air-drying and, as with the soil samples, stored at 76% relative humidity. After adding organic amendments to the soils, the mixtures were immediately shaken for 2 hours in an airtight glass container at room temperature (22°C), and prior to thermogravimetric analyses again conditioned at 76% relative air humidity for 2 weeks (*Tokarski* et al., 2018).

2.3 Incubation experiments

A RESPIROCOND-system (NORDGREEN INNOVATIONS, Sweden) with 96 vessels was used for the incubation experiment. The measurement of CO_2 is based on changed electrical conductivity of a 10 mL solution of 0.6 M potassium hydroxide by absorbing CO_2 in hermetically closed 250-mL vessels.

Depending on initial SOC content and expected level of CO_2 respiration, 20 or 40 g of soils of both sample sets (artificial soil mixtures and soils from LTAEs) in two analytical replications were rewetted under laboratory conditions to 60% of their field water retention capacity (determined at pF = 1.8) just before starting the monitoring of CO_2-respiration at a constant temperature of 25°C. Monitoring of CO_2-absorption started immediately after remoistening of soil samples and were repeated every 30 min with prolongation of the time interval to one measurement per hour during 120 days of incubation. The evaluation started with the entire data, but the measurements of the remoistened samples in the first 2.5 hours were excluded for better comparability after full temperature adjustment. It includes the transformation of the respiration rate into g CO_2-C per kg soil and g CO_2-C per g SOC.

2.4 Thermogravimetric analysis (TG)

The TG analyses were carried out in two analytical replicates for each sample before and after the incubation using a Mettler-Toledo TGA/SDTA 851e device. The air-dried soil sample of 0.8–1.0 g were placed in a ceramic pan and heated with a rate of 5°C min^{-1} from 25°C to 950°C. The sample mass was recorded every 4 seconds or one measurement for 0.3°C temperature increase. During the analytical procedure, the sample furnace was purged with an air stream enriched by 76% relative humidity at 22°C with a flow rate of ≈ 200 mL min^{-1}. The primary evaluation started with recalculation of mass losses to 1 g of soil and with reduction of data density to one mean thermal mass loss (TML) per 10°C temperature increase. The thermogravimetric profiles are presented as differential TG (DTG) curves, *i.e.*, the first derivative of the TML.TML is usually given with lower and upper temperature limit. For example, $TML_{290–300}$ describes TML from 290°C to 300°C.

2.5 Organic carbon content determination

The organic carbon (OC) contents of the artificial soil mixtures with organic amendments were determined in duplicate from the incubated and non-incubated samples by dry combustion with LECO TruMac CN analyser at an accredited laboratory (LKS mbH, Niederwiesa, Germany) using standard procedures (*ISO*, 1996).

2.6 Statistical analysis

At the beginning of the statistical analysis, nonparametric tests were performed to confirm the normal distribution and the lack of interdependence between the factors. Furthermore, a multifactorial ANOVA was carried out in order to consider the influence of two soil types (factor A), three types of organic amendments (factor B) before and after incubation (factor C) on measured parameters (incubation experiments: g CO_2-C per kg soil; thermal mass losses: mg per g soil in 10°C temperature increase; OC content: g C per kg soil). In addition, the results were verified by paired t-tests and modified ANOVA analyses.

The significance levels ($p < 0.001$, $p < 0.01$ or $p < 0.05$) are given in the text. The Tukey HSD test was applied to compare selected means from main and combination effects. Linear correlation analyses were performed to describe the relationships between TML and CO_2-C evolution. Presented significant data have a probability of at least of 95% ($R^2 > 0.6$). All statistical analyses were carried out using STATISTICA® software (version 12.0) or Microsoft Excel® (2013). All tests were repeated in R-statistics (version 3.2.1) for validation of the results.

3 Results

3.1 Incubation-induced changes in artificial soil mixtures with organic amendments

Figure 1 indicates increasing cumulative CO_2-C evolution with time in soils spiked with different organic amendments ($p < 0.001$ by Tukey-HSD test; Tab. 2a). The non-treated soils showed the lowest CO_2-C evolution without significant differences between sites ($p > 0.05$, data not shown) and without differences to samples spiked with CC and FYM. The CO_2-C evolution for all amendments increased in following order: non-treated soil ≤ CC ≤ FYM < SF < ST. A significant influence of the sites (clay content) was only detectable after the

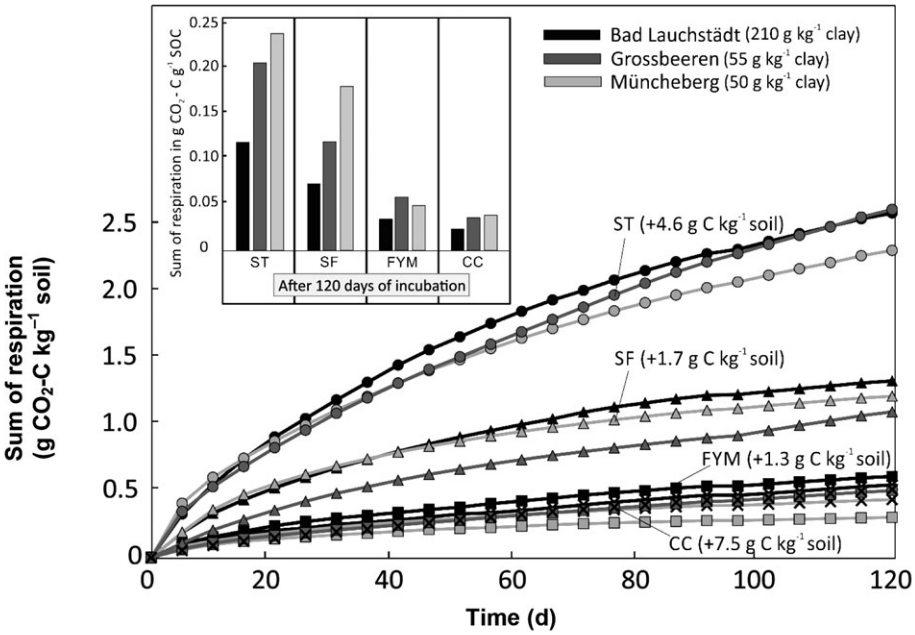

Figure 1: Dynamics of CO_2-C evolution per kg soil of non-fertilized soils in long-term field experiments in Bad Lauchstädt (black), Grossbeeren (dark grey), and Müncheberg (pale grey) each spiked with 60 Mg ha^{-1} fresh matter of straw (ST), sheep faeces (SF), farmyard manure (FYM), and charcoal (CC). The small inset shows the CO_2-C evolution per g SOC after 120 days of incubation.

Table 2: Multifactorial ANOVA for model experiments.[a]

(a) CO_2-C evolution (g g^{-1} SOC) after 120 days of incubation			
Factors	D.F.	*F*	*p*
Soil type (A)	2	64.0	0.000***
Organic amendments (B)	3	219.8	0.000***
Soil type × organic amendments (A × B)	6	11.2	0.000***
(b) Incubation-induced changes in soil organic carbon content			
Factors	D.F.	*F*	*p*
Soil type (A)	2	1580.6	0.000***
Organic amendments (B)	3	236.2	0.000***
Before/after incubation (C)	1	63.8	0.000***
Soil type × organic amendments (A × B)	6	0.5	0.826
Soil type × before/after incubation (A × C)	2	0.7	0.531
Organic amendments × before/after incubation (B × C)	3	14.7	0.000***
Soil type × organic amendments × before/after incubation (A × B × C)	6	0.4	0.842

[a]In the table are given: the degrees of freedom (D.F.), the variance ratio (F) and the probability level (p < 0.001***) to assess the changes of organic carbon content depending on soil, organic amendment and incubation. A: Bad Lauchstädt, Müncheberg and Grossbeeren, B: straw, farmyard manure, sheep faeces, charcoal, C: before and after 120-days incubation.

calculation of CO_2-C evolution per g SOC ($p < 0.001$, Tab. 2a). In this regard, higher CO_2-C evolution in soils of Müncheberg and Grossbeeren with added ST and SF was observed in comparison to Bad Lauchstädt (Fig. 1, small inset).

Figure 2 presents OC contents determined by elemental analyses before and after 120 days of incubation. As an example, soils in Bad Lauchstädt (BL) characterized by an initial SOC content of 16.1 g kg^{-1} (dotted black line). The application of ST (adding of 4.6 g C kg^{-1} soil) should increase the OC content approximately to 20.7 g C kg^{-1} soil. The measured OC content was 21.8 g C kg^{-1} soil (black column in Fig. 2; $p < 0.001$). The 120-day incubation of the soil with added ST decreased the OC content by 3.1 g C kg^{-1} soil to 18.7 g C kg^{-1} soil (dashed black column, $p < 0.001$). The increase in OC content after the addition of organic amendments was significant in all cases (*Tokarski* et al., 2018), while the reduction by incubation was significant only for ST, FYM and SF (Fig. 2, Tab. 2b).

The dynamics of TML of the non-treated soil in Bad Lauchstädt were distinguished from the soil in Müncheberg by higher TML (Fig. 3). This difference corresponds to differences in clay content and carbon accumulation of both soils. Adding straw increased the TML on both sites at temperatures between 250°C and 500°C and with a peak at ≈ 330°C ($p < 0.001$). Incubation reduced the TML in same temperature range with a peak at ≈ 330°C ($p < 0.01$). In temperature ranges below 200°C, peak shifts appear to be dependent on soil type. However, TML in these temperature ranges is not discussed as part of SOM in the literature, and the bound water in the air-dried soil samples has not been investigated. For this reason, these changes will not further discussed in this study.

The addition of organic amendments resulted mainly in changing dynamics of TML in temperature interval from 200°C to 500°C with individual peaks (Fig. 4a). For example, ST or SF showed a clear peak in TML at ≈ 330°C ($p < 0.01$), whereas FYM decayed continuously in a wide temperature range between 200 and 500°C. In contrast, CC application caused the highest increase of TML at higher temperature around 500°C. Incubation-induced changes are highest for ST and SF in the same temperature range as before incubation ≈ 330°C, $p < 0.01$;

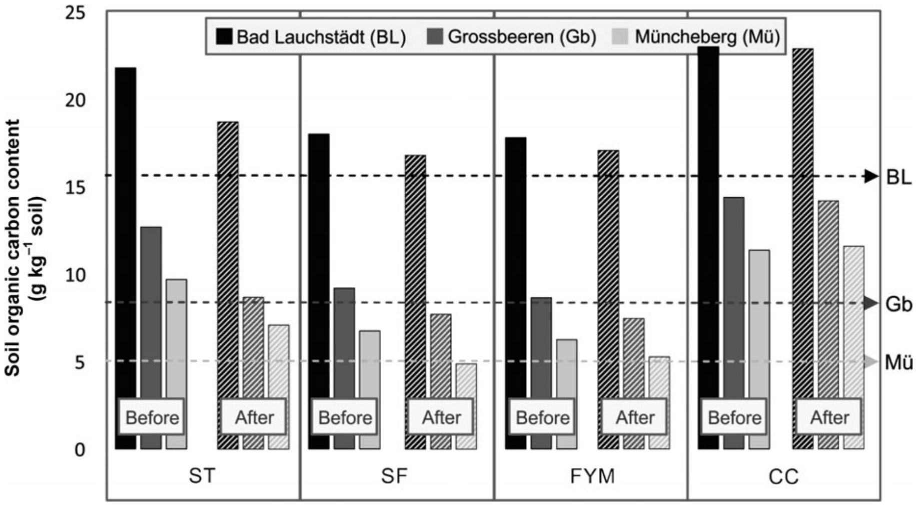

Figure 2: Organic carbon content in artificial mixtures of non-fertilized soils in long-term field experiments in Bad Lauchstädt (BL, black), Grossbeeren (GB, dark grey), and Müncheberg (Mü, pale grey) each spiked with 60 Mg ha^{-1} fresh matter of straw (ST), sheep faeces (SF), farmyard manure (FYM), and charcoal (CC) before and after 120 days of laboratory incubation. Horizontal dotted lines indicate the initial soil organic carbon contents of non-treated soils from LTAE-plots without fertilization.

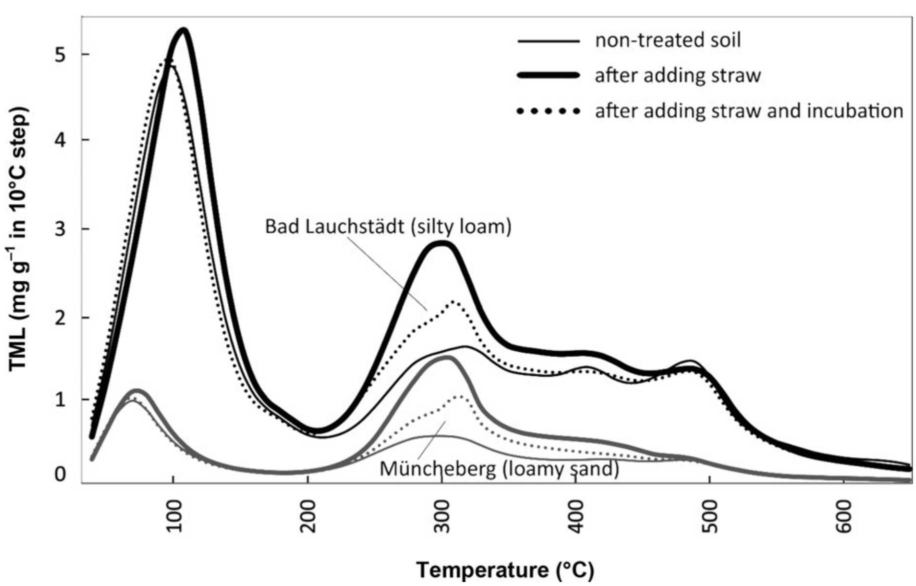

Figure 3: Dynamics of thermal mass losses (TML) for artificial mixtures of soils from selected sites with added straw (60 Mg ha^{-1} fresh matter or 4.6 g C kg^{-1} soil) before incubation (bold solid lines) and after incubation (dotted lines) compared to non-treated soils (thin solid lines).

Fig. 4b). FYM was degraded in larger temperature interval, but with a reduced mass loss intensity. The effects of CC were occurred mainly above 450°C (not significant).

In addition, correlation analyses of the artificial soil mixtures confirmed close relationships between the measured CO_2-C evolution and the incubation-induced changes in TML (differences in TML before and after incubation). For example, CO_2-C evolution during incubation experiments correlates with TML at 300°C (R^2 = 0.96) (similarly as in *Kučerík* and *Siewert*, 2014) and with TML at 410°C (R^2 = 0.95).

3.2 Incubation induced changes in soils from LTAEs

In all treatments with application of farmyard manure (FYM), we found the highest CO_2-C evolution ($p < 0.05$ by Tukey-HSD test) in both LTAEs in Bad Lauchstädt (Fig. 5a) and Müncheberg (Fig. 5b). Plant residues from corn (Bad Lauchstädt, V494) and weed flora succession (self-greening, Bad Lauchstädt, V505a) showed minor differences compared to FYM, but higher CO_2-C evolution when compared to non-fertilized and bare fallow treatments.

In Müncheberg, the treatments without fertilization and with mineral nitrogen (N) application indicated lowest values compared to samples with FYM ($p < 0.001$) and straw ($p < 0.05$ compared to non-fertilized plots only). Nevertheless, a site influence (clay content) was verified by higher values in Müncheberg compared to Bad Lauchstädt ($p < 0.001$, Fig. 5, small insets).

We found significantly larger changes in TML between 200 and 500°C for FYM with corn compared to bare fallow (Fig. 6a). Incubation had less influence on TML, which varied with the investigated temperature range. Therefore, minor changes occurred in treatments with FYM and corn in TML at ≈ 330°C (no significant), while a significant reduction after incubation can only be observed at ≈ 450°C ($p < 0.001$). Self-greening produced larger TML between 200 and 550°C (with a peak at ≈ 330°C) compared to mechanical bare fallow (Fig. 6b). Incubation led to a reduction in TML mainly at ≈ 330°C ($p < 0.01$) for self-greening. In treatments with mechanical fallow, the incubation-induced changes were particularly visible in TML at ≈ 450°C (increase, $p < 0.001$).

In Figure 7a, samples with FYM (black solid line) are characterized by larger TML between 250 and 450°C compared to non-fertilized treatments (dotted grey line). Incubation reduced the influence of FYM (Fig. 7b) in TML with two peaks at ≈ 390°C ($p < 0.01$) and at ≈ 450°C ($p < 0.05$). The addition of straw (grey solid line, Fig. 7a) did not cause an increase in TML compared to the non-fertilized treatments (dotted grey

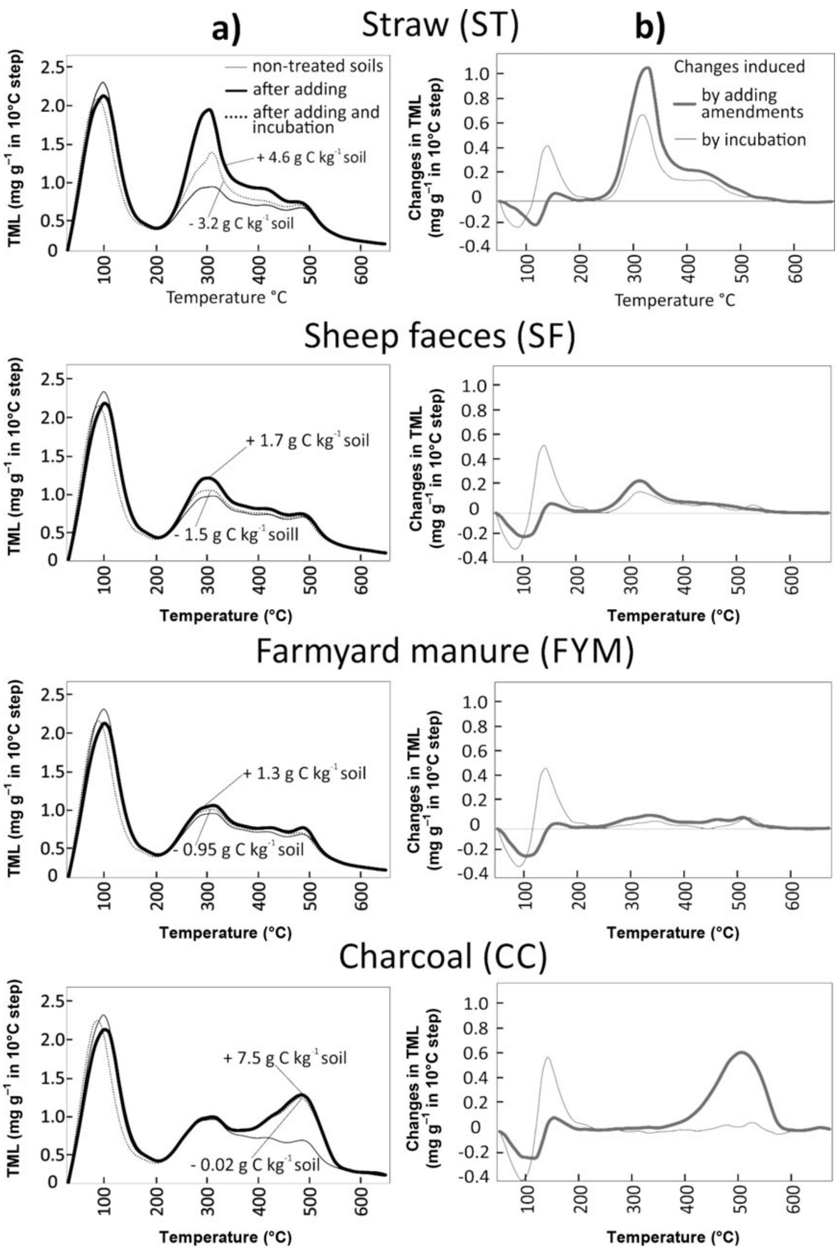

Figure 4: (a) Means of thermal mass losses (TML) of non-treated soils (black thin solid lines) presented as mean values of all sites (Bad Lauchstädt, Grossbeeren, Müncheberg) and artificial soil mixtures presented as mean values of all sites before incubation (black bold solid lines) and after incubation (black dotted lines). (b) Means of changes in dynamics of TML of all sites presented as differences between mixtures and non-treated soils before incubation (grey bold solid lines) and of artificial soil mixtures presented as differences between non-incubation and incubation (grey thin solid lines). Numbers show the amounts of added carbon to soils (+ value) and the mineralized carbon after 120 days of incubation (– value) presented as means of all sites

line, Fig. 7a), whereas the incubation reduced the TML at ≈ 380°C (p < 0.05) only (Fig. 7b). In case of N and non-fertilization, no significant changes in TML before and after incubation were found.

Correlation analyses of the soils from LTAEs revealed close relationships between CO_2-C evolution in incubation experiments and incubation-induced changes in TML. The highest linear coefficient of correlation was found for CO_2-C evolution with TML at 340°C (R^2 = 0.75), at 430°C (R^2 = 0.79) and for TML at 500°C (R^2 = 0.81).

4 Discussion

The estimated order in the increase of CO_2-C evolution after adding organic amendments to soils (artificial soil mixtures; Fig. 1) confirms literature data about the amendment-specific biodegradability in soils (charcoal < farmyard manure < sheep faeces < straw) (*Atkinson* et al., 2010; *Guenet* et al., 2010; *Grunwald* et al., 2016; *Hansen* et al., 2016; *Sarma* et al., 2017; *Teutscherova* et al., 2017; *Li* et al., 2018a). Aside from mineralization during incubation, the added organic amendments increased the OC content in soils (Tab. 2b). Dynamics of TML mirrored these changes in OC content caused by both adding amendments (*Tokarski* et al., 2018) and their incubation in soils (Figs. 2 and 3). Main differences in CO_2-C evolution and OC content between sites (LTAEs) can be explained with interdependency between content of SOC and clay (*Hassink*, 1997; *Körschens* and *Pfefferkorn*, 1998; *Barkusky*, 2009; *Rühlmann*, 2009; *Franko* and *Merbach*, 2017). Similar results can be found in the inset of Fig. 1, where microbiological degradation is expressed in incubation experiments in g CO_2-C per g SOC. However, a dependence of the biological degradation of the added organic amendments on the clay content of the soil is not proved. Because the CO_2-C evolution per kg soil was very similar at all sites (LTAEs, Fig. 1), we conclude that a clay-dependent mineralization from organic amendments can be concealed by the established clay-dependent organic carbon accumulation in soils or by carbon protection mechanisms. These considerations challenge the assessment of the biodegradability of SOM.

In the carried out experiments with both sample sets, the recording of thermal decay dynamics reveal changes in SOM by the addition of organic fertilizers to different soils in temperature ranges between 200 and 550°C. Microbiological degradation changes TML in same temperature ranges (Figs. 4, 6, and 7). This temperature area is known for thermal decomposition of organic matter (*Plante* et al., 2009; *Méndez* et al., 2013; *Pallasser* et al., 2013).

Biodegradability of organic amendments is a factor determining SOM quality and should be verifiable by incubation and thermogravimetric experiments. In our experiments, straw (in artificial soil mixtures) for example showed an effect on

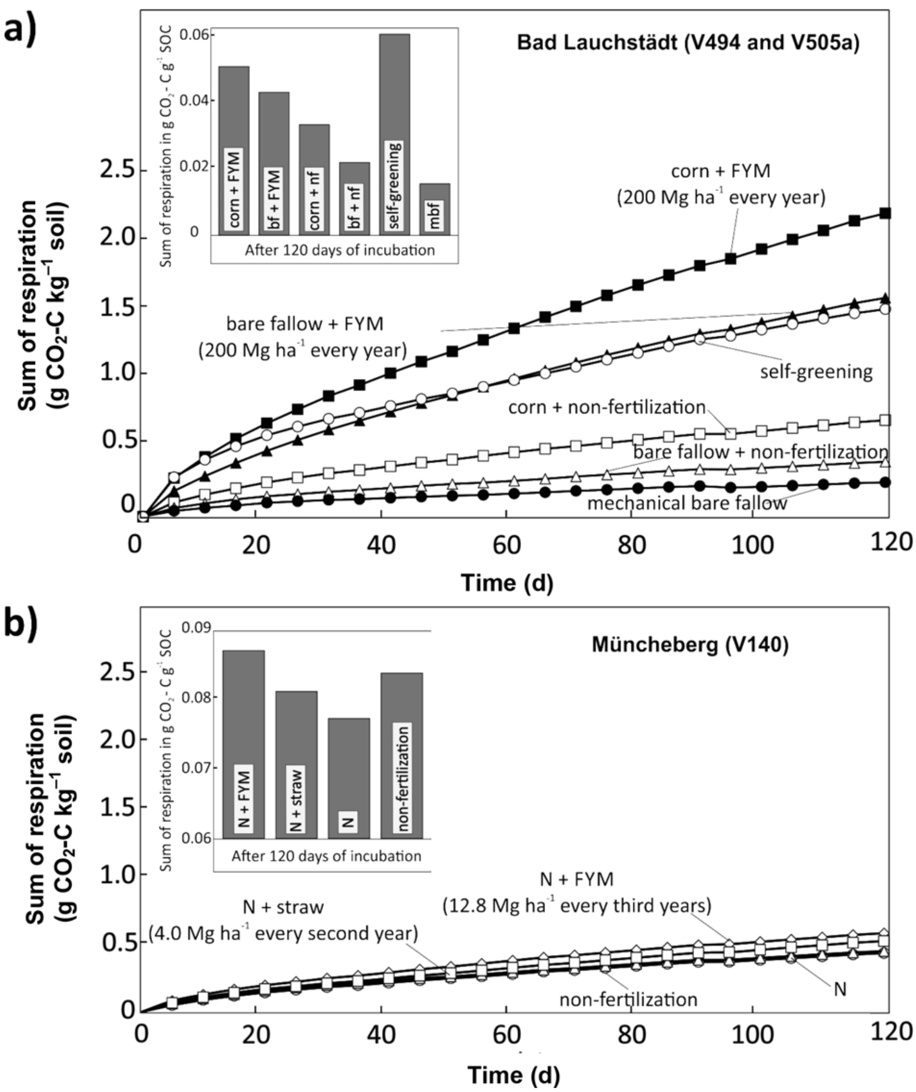

Figure 5: Dynamics of CO_2-C evolution per kg soil in incubation experiments with soils of different fertilization treatments from three long-term agricultural field experiments in (a) Bad Lauchstädt and (b) Müncheberg. The small inset shows the CO_2-C evolution per g SOC after 120 days of incubation. FYM = farmyard manure; bf = bare fallow; nf = non-fertilization; mbf = mechanical bare fallow; N = nitrogen.

CO_2-C evolution and incubation-induced changes in TML at ≈ 330°C. *Plante* et al. (2011) and *Siewert* et al. (2012) identified biodegradable SOM primarily in temperatures below 400°C. However, the incubation of soils of the LTAE in Müncheberg with long-term straw application (soils from LTAEs, Fig. 7) did not show large effects compared to straw in artificial soil mixtures. These results, however, can be explained with the high degradability of straw (*Powlson* et al., 2011, *Hansen* et al., 2016), with its low application rate (4 Mg ha^{-1} every second year), and with sampling of soils in the second year after straw application.

The LTAEs in Bad Lauchstädt did not contain any treatments with straw. However, corn and succession of weed flora (self-greening) seem to have similar impact as straw on both CO_2-C evolution and dynamics of TML with focus on temperatures ≈ 300°C (Fig. 6). If one considers the mineral nitrogen fertilization in the LTAE Müncheberg as a factor which promotes plant growth and the quantity of degradable plant residues in soils (*Gregorich* et al., 1996; *Mohanty* et al., 2013), similar changes can be observed as for straw (Fig. 7).

Summarizing these results, TML at ≈ 300°C seems to be an indicator for biodegradable organic residues in agricultural soils (*Kučerík* et al., 2013).

Furthermore, this indicator is close to TML for predicting carbon contents (*e.g*., TML320–330; *Siewert*, 2004), which reflects a clay-dependent carbon accumulation. For this reason, TML ≈ 300°C as an indicator for biodegradable components can be influenced by clay dependent organic carbon accumulation and requires a separate evaluation to distinguish freshly added carbon (organic amendments) from clay-dependent accumulated carbon.

The addition of sheep faeces (artificial soil mixtures) led to minor changes in CO_2-C evolution and in dynamics of TML compared to straw. This can be explained by lower quantity of added carbon and lower dry matter content (*Tokarski* et al., 2018). However, the incubation-induced changes in thermal decay dynamics were observed in larger temperature ranges with less pronounced focus on TML at ≈ 300°C. These specifics could indicate a mixture of both biodegradable (*e.g*., fresh forage) and stabilized organic components in result of its origin (*e.g*., digestion).

Farmyard manure in artificial soil mixtures caused even smaller changes in CO_2-C evolution compared to straw and sheep faeces. The repeated application of farmyard manure in LTAEs showed a higher influence with different impacts on CO_2-C evolution in Müncheberg and Bad Lauchstädt. A lower effect in artificial soil mixtures can be explained by the small quantity of added carbon and as a result of the lower dry matter content compared to straw, for example. The site-dependent influence on CO_2-C evolution of soils from LTAEs may be caused by the different quantities of added fertilizers (Bad Lauchstädt: 200 Mg ha^{-1} per year; Müncheberg: 12 Mg ha^{-1} every three years).

In both sample sets, changes in dynamics of TML caused by the addition and incubation of farmyard manure are located at the same temperature interval of 200 to 500°C. A missing

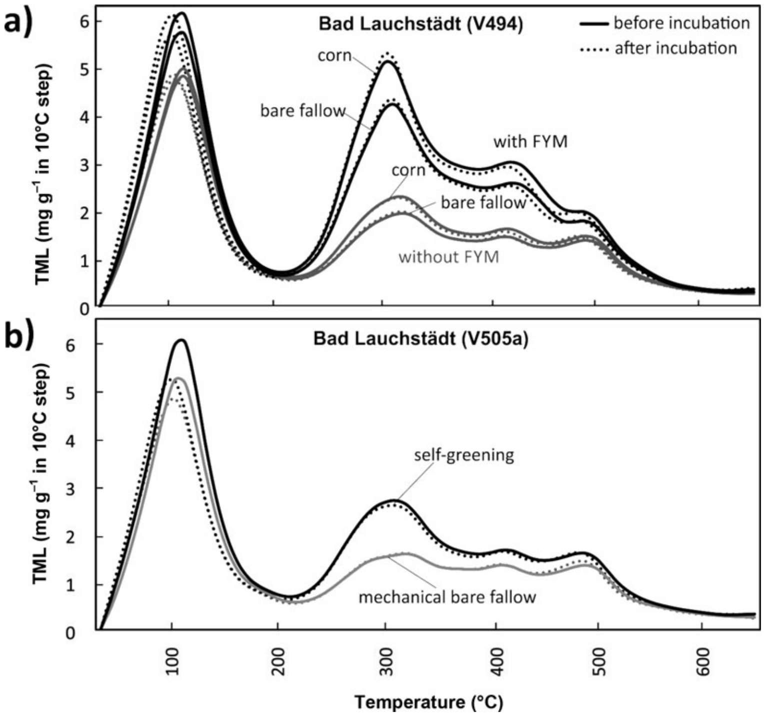

Figure 6: Dynamics of thermal mass losses (TML) for selected treatments of (a) V494 and (b) V505a in Bad Lauchstädt presented as mean values before (solid lines) and after incubation (dotted lines). FYM = farmyard manure.

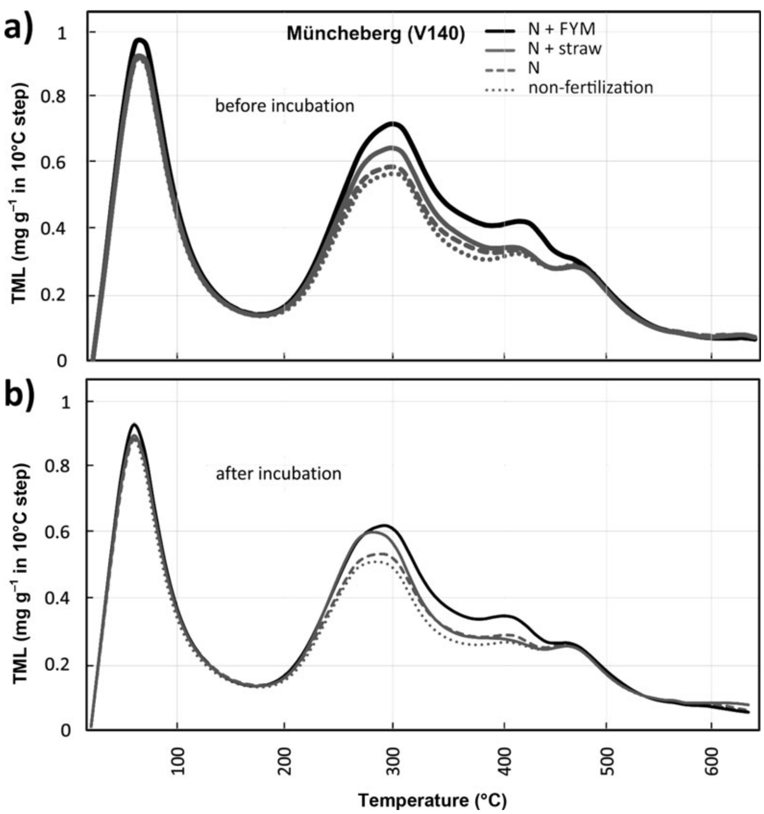

Figure 7: Dynamics of thermal mass losses (TML) for selected treatments of V140 in Müncheberg presented as mean values (a) before and (b) after incubation. FYM = farmyard manure, N = nitrogen.

emphasis on TML ≈ 300°C may result from a lower proportion of biodegradable components due to digestion and humification processes during storage of this fertilizer (*Ouatmane* et al., 2000; *Sánchez* et al., 2008). This special feature of farmyard manure reflects a better comparability with SOM characteristics compared to other organic fertilizers (*Haynes* and *Naidu*, 1998; *Lima* et al., 2009). This challenges the detection of farmyard manure using thermal decay dynamics (*Tokarski* et al., 2018) and underlines the necessity of distinguishing between different SOM components for their assessment in land use. It provokes again questions to what extent organic amendments and clay content influence carbon accumulation in soils (*Palm* et al., 2014).

Charcoal as an example for black carbon is distinguished from other organic amendments by higher thermal stability (*Leifeld*, 2007; *Plante* et al., 2009; *Cimò* et al., 2014) and low biological degradability (*Atkinson* et al., 2010; *Cayuela* et al., 2014; *Grunwald* et al., 2016; *Sarma* et al., 2017).

This particularity of charcoal explains the lack of significant changes in CO_2-C evolution notwithstanding the high quantity of added carbon. Nevertheless, charcoal seems to be traceable in artificial soil mixtures using TML above 450°C (*Tokarski* et al., 2018).

The different levels of correlation coefficients between CO_2-C evolution and TML for both artificial soil mixtures and soils from LTAEs can be explained by the differing proportion of added, degraded or accumulated organic components of SOM (*Kučerík* et al., 2016; *Tokarski* et al., 2018). Studies by *Peltre* et al. (2013) showed similar trends in changing diagnostic temperatures which were explained by different protective mechanisms of SOM and their interaction with soil minerals.

In summary, a combined evaluation of thermogravimetric analyses before and after incubation seems to provide information about biodegradation processes of SOM. However, overlapping TML's caused by organic amendments (*e.g.*, fresh residues or fertilizers) and SOM (clay-dependent accumulated carbon) requires adopted evaluation approaches. A possible approach could be the use of thermal mass losses in larger predefined temperature ranges (*Kučerík* et al., 2018) which can combine traditional fractionation of SOM with modern approaches such as density fractionation and mass spectroscopy for SOM quality determination (*Wiesmeier* et al., 2014; *Schiedung* et al., 2017).

5 Conclusion

In contrast to conclusions of *Schiedung* et al. (2017) about biological stability is not reflected in thermo-oxidative fractions, this study hints on a detectability of microbial degradation processes *via* recording of thermal mass losses in two independent experiments. However, these conclusions are based on methods for the assessment of thermal decay dynamics that go beyond the application of traditional peak analysis. Suitable reference objects are needed to compensate for the variety of unknown overlapping thermal degradation processes and to develop necessary evaluation algorithms. The conditioning of air-dried soil samples at comparable humidity and the content of bound water or clay in soil samples should be considered as a prerequisite to detect the interactions between thermal and biological degradability of SOM and its components. Such sample preparation may support the indirect distinction between soil organic carbon and organic carbon of fresh plant residues, organic fertilizers or other origin (*e.g*., charcoal) for the future assessment of SOM.

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References

Atkinson, C. J., *Fitzgerald, J. D.*, *Hipps, N. A.* (2010): Potential mechanisms for achieving agricultural benefits from biochar application to temperate soils: A review. *Plant Soil* 337, 1–18.

Barkusky, D. (2009): Müncheberger Nährstoffsteigerungsversuch, V140, in Ministerium für Ländliche Entwicklung, Umwelt und Verbraucherschutz, Landesamt für Verbraucherschutz, Landwirtschaft und Flurneuordnung (ed.): Dauerfeldversuche in Brandenburg und Berlin—Beiträge für eine nachhaltige landwirtschaftliche Bodenbenutzung, Brandenburg, Germany, pp. 103–109.

Barros, N., *Hansen, L. D.*, *Piñeiro, V.*, *Vikegard, P.* (2016): Calorimetry measures the response of soil organic matter biodegradation to increasing temperature. *J. Therm. Anal. Calorim.* 123, 2397–2403.

Bending, G. D., *Turner, M. K.* (1999): Interaction of biochemical quality and particle size of crop residues and its effect on the microbial biomass and nitrogen dynamics following incorporation into soil. *Biol. Fertil. Soils* 29, 319–327.

Cayuela, M. L., *van Zwieten, L.*, *Singh, B. P.*, *Jeffery, S.*, *Roig, A.*, *Sánchez-Monedero, M. A.* (2014): Biochar's role in mitigating soil nitrous oxide emissions: A review and meta-analysis. *Agric. Ecosyst. Environ.* 191, 5–16.

Cimò, G., *Kučerík, J.*, *Berns, A. E.*, *Schaumann, G. E.*, *Alonzo, G.*, *Conte, P.* (2014): Effect of heating time and temperature on the chemical characteristics of biochar from poultry manure. *J. Agr. Food Chem.* 62, 1912–1918.

Conant, R. T., *Ryan, M. G.*, *Ågren, G. I.*, *Birge, H. E.*, *Davidson, E. A.*, *Eliasson, P. E.*, *Evans, S. E.*, *Frey, S. D.*, *Giardina, C. P.*, *Hopkins, F. M.*, *Hyvönen, R.*, *Kirschbaum, M. U. F.*, *Lavallee, J. M.*, *Leifeld, J.*, *Parton, W. J.*, *Megan Steinweg, J.*, *Wallenstein, M. D.*, *Wetterstedt, M. J. Å.*, *Bradford, M. A.* (2011): Temperature and soil organic matter decomposition rates – synthesis of current knowledge and a way forward. *Global Change Biol.* 17, 3392–3404.

Dignac, M. F., *Derrien, D.*, *Barré, P.*, *Barot, S.*, *Cécillon, L.*, *Chenu, C.*, *Chevallier, T.*, *Freschet, G. T.*, *Garnier, P.*, *Guenet, B.*, *Hedde, M.*, *Klumpp, K.*, *Lashermes, G.*, *Maron, P. A.*, *Nunan, N.*, *Roumet, C.*, *Basile-Doelsch, I.* (2017): Increasing soil carbon storage: mechanisms, effects of agricultural practices and proxies. A review. *Agron. Sustain. Dev.* 37. DOI: https://doi.org/10.1007/s13593-017-0421-2.

Fernández-Gálvez, J., *Gálvez, A.*, *Peña, A.*, *Mingorance, M. D.* (2012): Soil hydrophysical properties resulting from the interaction between organic amendments and water quality in soils from Southeastern Spain—a laboratory experiment. *Agr. Water Manage.* 104, 104–112.

Ferreras, L., *Gómez, E.*, *Toresani, S.*, *Firpo, I.*, *Rotondo, R.* (2006): Effect of organic amendments on some physical, chemical and biological properties in a horticultural soil. *Bioresour. Technol.* 97, 635–640.

Field, C. B., *Lobell, D. B.*, *Peters, H. A.*, *Chiariello, N. R.* (2007): Feedbacks of terrestrial ecosystems to climate change. *Annu. Rev. Environ. Resour.* 32, 1–29.

Franko, U., *Merbach, I.* (2017): Modelling soil organic matter dynamics on a bare fallow Chernozem soil in Central Germany. *Geoderma* 303, 93–98.

Glaser, B., *Lehmann, J.*, *Zech, W.* (2002): Ameliorating physical and chemical properties of highly weathered soils in the tropics with charcoal—a review. *Biol. Fertil. Soils* 35, 219–230.

Gregorich, E. G., *Gillespie, A. W.*, *Beare, M. H.*, *Curtin, D.*, *Sanei, H.*, *Yanni, S. F.* (2015): Evaluating biodegradability of soil organic matter by its thermal stability and chemical composition. *Soil Biol. Biochem.* 91, 182–191.

Gregorich, E. G., *Liang, B. C.*, *Ellert, B. H.*, *Drury, C. F.* (1996): Fertilization effects on soil organic matter turnover and corn residue C storage. *Soil Sci. Soc. Am. J.* 60, 472–476.

Grunwald, D., *Kaiser, M.*, *Ludwig, B.* (2016): Effect of biochar and organic fertilizers on C mineralization and macro-aggregate dynamics under different incubation temperatures. *Soil Till. Res.* 164, 11–17.

Guenet, B., *Neill, C.*, *Bardoux, G.*, *Abbadie, L.* (2010): Is there a linear relationship between priming effect intensity and the amount of organic matter input? *Appl. Soil Ecol.* 46, 436–442.

Hansen, V., *Müller-Stöver, D.*, *Munkholm, L. J.*, *Peltre, C.*, *Hauggaard-Nielsen, H.*, *Jensen, L. S.* (2016): The effect of straw and wood gasification biochar on carbon sequestration, selected soil fertility indicators and functional groups in soil: an incubation study. *Geoderma* 269, 99–107.

Hassink, J. (1997): The capacity of soils to preserve organic C and N by their association with clay and silt particles. *Plant Soil* 191, 77–87.

Haynes, R. J., *Naidu, R.* (1998): Influence of lime, fertilizer and manure applications on soil organic matter content and soil physical conditions: a review. *Nutr. Cycl. Agroecosyst.* 51, 123–137.

Hopkins, D. W. (2007): Carbon Mineralization, in Carter, M. R., Gregorich, E. G. (eds.): Soil Sampling and Methods of Analysis, 2nd Ed. Canadian Society of Soil Science, Taylor and Francis Group, Abingdon, UK, pp. 589–598.

ISO—International Organization for Standardization (1996): Soil Quality—Determination of Organic and Total Carbon After Dry Combustion (Elementary Analysis) (ISO 10694:1995). Beuth Verlag GmbH, Berlin, Germany.

Jin, J., *Kang, M.*, *Sun, K.*, *Pan, Z.*, *Wu, F.*, *Xing, B.* (2016): Properties of biochar-amended soils and their sorption of imidacloprid, isoproturon, and atrazine. *Sci. Total Environ.* 550, 504–513.

Johnson, J. M.-F., *Barbour, N. W.*, *Weyers, S. L.* (2007): Chemical composition of crop biomass impacts its decomposition. *Soil Sci. Soc. Am. J.* 71, 155–162.

Kätterer, T., *Börjesson, G.*, *Kirchmann, H.* (2014): Changes in organic carbon in topsoil and subsoil and microbial community composition caused by repeated additions of organic amendments and N fertilisation in a long-term field experiment in Sweden. *Agric. Ecosyst. Environ.* 189, 110–118.

Körschens, M., *Albert, E.*, *Armbruster, M.*, *Barkusky, D.*, *Baumecker, M.*, *Behle-Schalk, L.*, *Bischoff, R.*, *Čergan, Z.*, *Ellmer, F.*, *Herbst, F.*, *Hoffmann, S.*, *Hofmann, B.*, *Kismanyoky, T.*, *Kubat, J.*, *Kunzova, E.*, *Lopez-Fando, C.*, *Merbach, I.*, *Merbach, W.*, *Pardor, M. T.*, *Rogasik, J.*, *Rühlmann, J.*, *Spiegel, H.*, *Schulz, E.*, *Tajnsek, A.*, *Toth, Z.*, *Wegener, H.*, *Zorn, W.* (2013): Effect of mineral and organic fertilization on crop yield, nitrogen uptake, carbon and nitrogen balances, as well as soil organic carbon content and dynamics: results from 20 European long-term field experiments of the twenty-first century. *Arch. Agron. Soil Sci.* 59, 1017–1040.

Körschens, M., *Pfefferkorn, A.* (1998): Der Statische Düngungsversuch und andere Feldversuche [The Static Fertilization Experiment and other Long-Term Field Experiments]. UFZ-Umweltforschungszentrum Leipzig-Halle GmbH, Halle, Germany.

Körschens, M., *Schulz, E.*, *Knappe, S.* (1994): Einfluss von Dauerbrache und Fruchtfolge auf die N-Bilanzen einer Löss-Schwarzerde unter Berücksichtigung extremer Düngungsvarianten. *Arch. Agron. Soil Sci.* 38, 415–422.

Kristl, M., *Muršec, M.*, *Šuštar, V.*, *Kristl, J.* (2016): Application of thermogravimetric analysis for the evaluation of organic and inorganic carbon contents in agricultural soils. *J. Therm. Anal. Calorim.* 123, 2139–2147.

Kučerík, J., *Čtvrtníčková, A.*, *Siewert, C.* (2013): Practical application of thermogravimetry in soil science: Part 1. Thermal and biological stability of soils from contrasting regions. *J. Therm. Anal. Calorim.* 113, 1103–1111.

Kučerík, J., *Siewert, C.* (2014): Practical application of thermogravimetry in soil science: Part 2. Modeling and prediction of soil respiration using thermal mass losses. *J. Therm. Anal. Calorim.* 116, 563–570.

Kučerík, J., *Demyan, M. S.*, *Siewert, C.* (2016): Practical application of thermogravimetry in soil science: Part 4. Relationship between clay, organic carbon and organic matter contents. *J. Therm. Anal. Calorim.* 123, 2441–2450.

Kučerík, J., *Tokarski, D.*, *Demyan, M. S.*, *Merbach, I.*, *Siewert, C.* (2018): Linking soil organic matter thermal stability with contents of clay, bound water, organic carbon and nitrogen. *Geoderma* 316, 38–46.

Kuzyakov, Y., *Subbotina, I.*, *Chen, H.*, *Bogomolova, I.*, *Xu, X.* (2009): Black carbon decomposition and incorporation into soil microbial biomass estimated by ^{14}C labeling. *Soil Biol. Biochem.* 41, 210–219.

Leifeld, J. (2007): Thermal stability of black carbon characterised by oxidative differential scanning calorimetry. *Org. Geochem.* 38, 112–127.

Li, J., *Wen, Y.*, *Li, X.*, *Li, Y.*, *Yang, X.*, *Lin, Z.*, *Song, Z.*, *Cooper, J. M.*, *Zhao, B.* (2018a): Soil labile organic carbon fractions and soil organic carbon stocks as affected by long-term organic and mineral fertilization regimes in the North China Plain. *Soil Till. Res.* 175, 281–290.

Li, Y., *Hu, S.*, *Chen, J.*, *Müller, K.*, *Li, Y.*, *Fu, W.*, *Lin, Z.*, *Wang, H.* (2018b): Effects of biochar application in forest ecosystems on soil properties and greenhouse gas emissions: a review. *J. Soil. Sediment.* 18, 546–563.

Lima, D. L. D., *Santos, S. M.*, *Scherer, H. W.*, *Schneider, R. J.*, *Duarte, A. C.*, *Santos, E. B. H.*, *Esteves, V. I.* (2009): Effects of organic and inorganic amendments on soil organic matter properties. *Geoderma* 150, 38–45.

Lopez-Capel, E., *Sohi, S. P.*, *Gaunt, J. L.*, *Manning, D. A. C.* (2005): Use of thermogravimetry–Differential Scanning Calorimetry to Characterize Modelable Soil Organic Matter Fractions. *Soil Sci. Soc. Am. J.* 69, 136–140.

Méndez, A., *Tarquis, A. M.*, *Saa-Requejo, A.*, *Guerrero, F.*, *Gascó, G.* (2013): Influence of pyrolysis temperature on composted sewage sludge biochar priming effect in a loamy soil. *Chemosphere* 93, 668–676.

Mohanty, M., *Reddy, K. S.*, *Probert, M. E.*, *Dalal, R. C.*, *Rao, A. S.*, *Menzies, N. W.* (2011): Modelling N mineralization from green manure and farmyard manure from a laboratory incubation study. *Ecol. Modell.* 222, 719–726.

Mohanty, S., *Nayak, A. K.*, *Kumar, A.*, *Tripathi, R.*, *Shahid, M.*, *Bhattacharyya, P.*, *Raja, R.*, *Panda, B. B.* (2013): Carbon and nitrogen mineralization kinetics in soil of rice? Rice system under long term application of chemical fertilizers and farmyard manure. *Eur. J. Soil Biol.* 58, 113–121.

Nguyen, T. T., *Cavagnaro, T. R.*, *Thanh Ngo, H. T. T.*, *Marschner, P.* (2016): Soil respiration, microbial biomass and nutrient availability in soil amended with high and low C/N residue—Influence of interval between residue additions. *Soil Biol. Biochem.* 95, 189–197.

Otero, M., *Calvo, L. F.*, *Estrada, B.*, *García, A. I.*, *Morán, A.* (2002): Thermogravimetry as a technique for establishing the stabilization progress of sludge from wastewater treatment plants. *Thermochim. Acta* 389, 121–132.

Ouatmane, A., *Provenzano, M. R.*, *Hafidi, M.*, *Senesi, N.* (2000): Compost maturity assessment using calorimetry, spectroscopy and chemical analysis. *Compost Sci. Util.* 8, 124–134.

Pallasser, R., *Minasny, B.*, *McBratney, A. B.* (2013): Soil carbon determination by thermogravimetrics. *PeerJ* 1. DOI: https://doi.org/10.7717/peerj.6.

Palm, C., *Blanco-Canqui, H.*, *DeClerck, F.*, *Gatere, L.*, *Grace, P.* (2014): Conservation agriculture and ecosystem services: An overview. *Agric., Ecosyst. Environ.* 187, 87–105.

Paul, E. A., *Morris, S. J.*, *Conant, R. T.*, *Plante, A. F.* (2006): Does the acid hydrolysis–incubation method measure meaningful soil organic carbon pools? *Soil Sci. Soc. Am. J.* 70, 1023–1035.

Peltre, C., *Fernández, J. M.*, *Craine, J. M.*, *Plante, A. F.* (2013): Relationships between biological and thermal indices of soil organic matter stability differ with soil organic carbon level. *Soil Sci. Soc. Am. J.* 77, 2020–2028.

Plante, A. F., *Fernández, J. M.*, *Haddix, M. L.*, *Steinweg, J. M.*, *Conant, R. T.* (2011): Biological, chemical and thermal indices of soil organic matter stability in four grassland soils. *Soil Biol. Biochem.* 43, 1051–1058.

Plante, A. F., *Fernández, J. M.*, *Leifeld, J.* (2009): Application of thermal analysis techniques in soil science. *Geoderma* 153, 1–10.

Plante, A. F., *Pernes, M.*, *Chenu, C.* (2005): Changes in clay-associated organic matter quality in a C depletion sequence as measured by differential thermal analyses. *Geoderma* 129, 186–199.

Powlson, D. S., *Glendining, M. J.*, *Coleman, K.*, *Whitmore, A. P.* (2011): Implications for Soil Properties of Removing Cereal Straw: Results from Long-Term Studies. *Agron. J.* 103, 279–287.

Prescott, C. E. (2010): Litter decomposition: What controls it and how can we alter it to sequester more carbon in forest soils? *Biogeochemistry* 101, 133–149.

Pronk, G. J., *Heister, K.*, *Kögel-Knabner, I.* (2013): Is turnover and development of organic matter controlled by mineral composition? *Soil Biol. Biochem.* 67, 235–244.

Rühlmann, J. (2009): Statistischer Dauerfeldversuch "Organisch-mineralische N-Düngung" Grossbeeren (1989), in Ministerium für Ländliche Entwicklung, Umwelt und Verbraucherschutz, Landesamt für Verbraucherschutz, Landwirtschaft und Flurneuordnung (ed.): Dauerfeldversuche in Brandenburg und Berlin—Beiträge für eine nachhaltige landwirtschaftliche Bodenbenutzung. IGZ, Brandenburg, Germany, pp. 73–86.

Sánchez, M., *Gomez, X.*, *Barriocanal, G.*, *Cuetos, M. J.*, *Morán, A.* (2008): Assessment of the stability of livestock farm wastes treated by anaerobic digestion. *Int. Biodeterior. Biodegrad.* 62, 421–426.

Sarma, B., *Borkotoki, B.*, *Narzari, R.*, *Kataki, R.*, *Gogoi, N.* (2017): Organic amendments: Effect on carbon mineralization and crop productivity in acidic soil. *J. Cleaner Prod.* 152, 157–166.

Schiedung, H., *Bauke, S.*, *Bornemann, L.*, *Welp, G.*, *Borchard, N.*, *Amelung, W.* (2016): A simple method for in-situ assessment of soil respiration using alkali absorption. *Appl. Soil Ecol.* 106, 33–36.

Schiedung, M., *Don, A.*, *Wordell-Dietrich, P.*, *Alcántara, V.*, *Kuner, P.*, *Guggenberger, G.* (2017): Thermal oxidation does not fractionate soil organic carbon with differing biological stabilities. *J. Plant Nutr. Soil Sci.* 180, 18–26.

Schröder, J. J., *Schulte, R. P. O.*, *Creamer, R. E.*, *Delgado, A.*, *van Leeuwen, J.*, *Lehtinen, T.*, *Rutgers, M.*, *Spiegel, H.*, *Staes, J.*, *Tóth, G.*, *Wall, D. P.*, *de Varennes, A.* (2016): The elusive role of soil quality in nutrient cycling: A review. *Soil Use Manage.* 32, 476–486.

Shen, W., *Reynolds, J. F.*, *Hui, D.* (2009): Responses of dryland soil respiration and soil carbon pool size to abrupt vs. gradual and individual vs. combined changes in soil temperature, precipitation, and atmospheric [CO 2]: A simulation analysis. *Global Change Biol.* 15, 2274–2294.

Siewert, C. (2001): Investigation of the thermal and biological stability of soil organic matter. Habilitation, Technical University Berlin, Germany.

Siewert, C. (2004): Rapid screening of soil properties using thermogravimetry. *Soil Sci. Soc. Am. J.* 68, 1656–1661.

Siewert, C., *Demyan, M. S.*, *Kučerík, J.* (2012): Interrelations between soil respiration and its thermal stability. *J. Therm. Anal. Calorim.* 110, 413–419.

Siewert, C., *Kučerík, J.* (2015): Practical applications of thermogravimetry in soil science—Part 3: I interrelations between soil components and unifying principles of pedogenesis. *J. Therm. Anal. Calorim.* 120, 471–480.

Soucémarianadin, L., *Cécillon, L.*, *Chenu, C.*, *Baudin, F.*, *Nicolas, M.*, *Girardin, C.*, *Barré, P.* (2018): Is Rock-Eval 6 thermal analysis a good indicator of soil organic carbon lability?—A method-comparison study in forest soils. *Soil Biol. Biochem.* 117, 108–116.

Teutscherova, N., *Vazquez, E.*, *Santana, D.*, *Navas, M.*, *Masaguer, A.*, *Benito, M.* (2017): Influence of pruning waste compost maturity and biochar on carbon dynamics in acid soil: Incubation study. *Eur. J. Soil Biol.* 78, 66–74.

Tokarski, D., *Kučerík, J.*, *Kalbitz, K.*, *Demyan, M. S.*, *Merbach, I.*, *Barkusky, D.*, *Ruehlmann, J.*, *Siewert, C.* (2018): Contribution of organic amendments to soil organic matter detected by thermogravimetry. *J. Plant Nutr. Soil Sci.* 181, 664–674.

Totsche, K. U., *Rennert, T.*, *Gerzabek, M. H.*, *Kögel-Knabner, I.*, *Smalla, K.*, *Spiteller, M.*, *Vogel, H.-J.* (2010): Biogeochemical interfaces in soil: The interdisciplinary challenge for soil science. *J. Plant Nutr. Soil Sci.* 173, 88–99.

von Lützow, M. V., *Kogel-Knabner, I.*, *Ekschmitt, K.*, *Matzner, E.*, *Guggenberger, G.*, *Marschner, B.*, *Flessa, H.* (2006): Stabilization of organic matter in temperate soils: Mechanisms and their relevance under different soil conditions—a review. *Eur. J. Soil Sci.* 57, 426–445.

Weil, R. W., *Brady, N. C.* (2016): The Nature and Properties of Soils. Pearson/Prentice Hall, Upper Saddle River, NJ, USA.

Wiesmeier, M., *Schad, P.*, *von Lützow, M.*, *Poeplau, C.*, *Spörlein, P.*, *Geuß, U.*, *Hangen, E.*, *Reischl, A.*, *Schilling, B.*, *Kögel-Knabner, I.* (2014): Quantification of functional soil organic carbon pools for major soil units and land uses in southeast Germany (Bavaria). *Agric. Ecosyst. Environ.* 185, 208–220.

Xu, Y., *Chen, Z.*, *Ding, W.*, *Fan, J.* (2017): Responses of manure decomposition to nitrogen addition: Role of chemical composition. *Sci. Total Environ.* 587, 11–21.

