

# Linking soil organic matter thermal stability with contents of clay, bound water, organic carbon and nitrogen

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## ABSTRACT

Thermogravimetry is a technique measuring mass change during programmed heating. In soil analysis, it is used for determination of content of volatile fractions, thermally labile and stable fractions of soil organic matter and minerals. One method of data analysis uses the determination of mass losses in 10 °C temperature areas. In the past, their mutual correlation revealed several larger temperature areas of mass losses, which appeared to be universal for all types of soils equilibrated at the same relative humidity. However, it is unclear if mass losses in these temperature areas are connected with biogeochemical functions or processes in soil. In this work, using data from > 300 soils of different types, geographical origin, and land uses we demonstrate their linear correlation with content of organic C, total N and clay content and biological activity or their combinations. In particular, the results showed that mass losses between 200 and 300 °C, which is related to thermally labile fraction, correlates best with total organic C and less with total organic N. From 300 to 450 °C represents a more stabilized soil organic matter pool and can be best described by either by C or N contents. Mass loss 450–550 °C correlates strongly with clay content, which suggests a connection to organo-clay complexes. The low temperature interval of 30–200 °C, which corresponded to weakly and strongly bound water showed a strong connection with clay content, but a weaker connection to microbial activity. The developed equations were corrected and verified using additional soil sample sets. The correlations along with their universal applicability lead to conclusion about the possible connection of mass losses in these temperature areas to, still unknown, biogeochemical soil functions. The obtained equations may represent a new approach of rapid and universally applicable “mathematical” fractionation requiring only contents of soil organic carbon, total nitrogen, clay and water in soil equilibrated at 76% relative humidity.

## 1. Introduction

“Soil is a complex system composed of a set of interconnected physical, chemical and biological factors that function as a whole” (Minasny et al., 2008). Primarily, soil heterogeneity and complexity is caused by soil forming factors being climate, topography, geological parent material, biota, time (Jenny, 1994) and anthropogenic influences. The interplay between forming factors gives rise to a high variability of soil forming processes and results in formation of different soil types covering a wide range of properties and composition. The variation and interactions between soil forming factors are believed to be the reason for the absence of a generic model useful for the

prediction of properties in a wide range of soils (Schmidt et al., 2011). However, many soils share similar features in their composition (e.g. C/N content, amino acids composition, phenotypic and genotypic characteristics of soil microbial communities (Fierer et al., 2009)) and these may be used for the development of a universally applicable model for the prediction of soil properties irrespective their origin and composition (Siewert and Kucerik, 2015).

Soil organic matter (SOM) is a key parameter that determines quality, productivity and ecological functioning of soils. SOM contains a large portion of the global terrestrial carbon pool, and for this reason soils are currently being discussed for their capacity to offset increasing levels of atmospheric CO<sub>2</sub> (Schmidt et al., 2011). This is reflected in the

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emphasis in developing methods for rapid assessment of SOM turnover rates, sizes and composition of different C pools and also to better understand SOM protection mechanisms (Pallasser et al., 2013).

Methods of thermal analysis are gradually gaining interest in soils science. Generally, they belong to the family of techniques that use heat to measure properties of soil exposed to a temperature program. One of the most frequently used techniques is calorimetry (both isothermal and non-isothermal) which is based on measurement of temperature that gives enthalpy upon instrument calibration. In soil science calorimetry techniques have been used to evaluate the quality of organic matter (Barros et al., 2011b; Plante et al., 2005), insight into carbon cycling (Herrmann et al., 2014), soil microbiological activity (Barros et al., 2011a; Barros et al., 2015; Barros et al., 2007; Barros et al., 2010), stability of water molecule bridges (Ondruch et al., 2017b; Schaumann and LeBoeuf, 2005), properties of soil aliphatic crystallites (Ondruch et al., 2017a), role of cations in soil organic matter stability (Diehl et al., 2014) or phase transitions in soil organic matter (LeBoeuf and Zhang, 2009; Young and LeBoeuf, 2000).

Another widely used thermoanalytical technique is thermogravimetry (TG), which is based on the determination of mass change of soil at either non-isothermal or isothermal conditions. For soil analysis, mostly linear heating programs are used (Fernández et al., 2011). As a result, TG analyzes thermal or thermo-oxidative stability of soils, which is determined mainly by binding energies, accessibility of SOM components, their mutual interactions and other factors (Plante et al., 2009). TG provides both quantitative (mass loss) and qualitative (temperature) information. Alone, or in a combination with other detectors, TG has been used to determine the ratio between thermally labile and stable soil organic matter (Peikert et al., 2015; Tamimi et al., 2017), stability of soil organic matter components (Manning et al., 2005), the shift in stability of soil organic matter supramolecular complexes induced by small organic molecules and cations (Buurman et al., 2002), the content (De la Rosa et al., 2008; Lopez-Capel et al., 2006) and presence (Siewert and Kucerik, 2015) of pyrogenic carbon, stability of organic matter fractions such as humic acids (Rotaru et al., 2008) or for distinguishing of organic and inorganic carbon (Kristl et al., 2016).

Traditional TG data evaluation employs a temperature or time derivative TG curve (DTG). The local minima in the DTG curve are interpreted as a change in degradation mechanisms reflecting the change in composition of degraded material. To illustrate it, Fig. 1 reports the TG curves (black line) of soil investigated in this work and DTG (bold grey line) and an alternative way of data analysis discussed further on.

To avoid soil-dependent differences, an alternative approach of TG data evaluation representing the determination of mass losses in predefined temperature intervals is sometimes used e.g. (Kristl et al., 2016). An alternative way of this approach (Siewert, 2001) suggests the use of mass losses in 10 °C temperature intervals (here referred as TML)

of air dried soils equilibrated at constant relative humidity (RH). This approach is based on the view that progressive mass loss determined by using TG is a separation of soil parts according to their stability, and separation of mass losses into 10 °C intervals represent a good way to minimize the data number while keeping reproducibility of mass loss determination. The constant RH is used to determine the mass losses under thermodynamically comparable conditions, i.e. soils are in equilibrium with their environment under the same moisture conditions. Up to now, this approach was tested only for soils equilibrated at 76% RH and lead to the discovery of connections between TML and soil properties such as soil organic carbon (SOC), total nitrogen content (TN), clay content (Siewert, 2001; Siewert, 2004), and microbiological activity indicators reflected in amount of CO<sub>2</sub> released by soil microorganisms (Kucerik et al., 2013; Kucerik and Siewert, 2014; Siewert et al., 2012). The most important TML, which will be further discussed in this work and their characteristics and methods of determination together with coefficients of determination are reported in Table 1. Furthermore, the combination of TMLs has been demonstrated to correlate with total SOM content represented by mass losses from 110 to 550 °C (referred here as LTML<sub>110–550</sub>). The correlation enabled the development of equations for SOM determination using soil organic C (or total N) and clay for a wide range of soil types from various climatic zones and soil management (Kucerik et al., 2016). The deviation from the equations seems to be useful to distinguish fresh, undecomposed organic material from more highly decomposed soil organic matter (Kucerik et al., 2016). Next, correlating all TMLs with each other (autocorrelation method) in the temperature interval 30–950 °C revealed several temperature areas in which TMLs significantly correlated. For better understanding, we report an exemplary raster graph showing the coefficients of determination as grey shades (Fig. 2). It is hypothesized that these temperature areas indicate the presence of molecules or soil parts, which are mutually interconnected either directly or as a result of biotic and/or abiotic soil forming processes (Siewert and Kucerik, 2015). The temperature areas are marked as A–F in Fig. 2 and are also summarized in Table 2. Briefly, the areas A (30–100 °C) and B (100–200 °C) represent loosely and strongly bound water, respectively, C (200–300 °C), D (300–450 °C), E (450–550 °C) are temperature areas representing fractions of soil organic matter (and possibly pyrogenic carbon) of increasing thermal stability and G (above 550 °C) represents soil inorganic matter. F is the combination of the C, D and E temperature areas. It is important to note that the mass loss in F area is also caused by elimination of water bound in clay minerals, which has implications to understanding to soil forming processes (Kucerik et al., 2016).

The comparison of the traditional DTG-based approach with TML-based approach reveals that the minima in DTG are soil dependent, therefore, this approach is applicable to distinguish differences in soil composition (Peikert et al., 2015). On the contrary, the temperature areas A–F obtained from TML-based approach are the same for different soil types, which indicates generic similarities in composition of soils and their stability fractions (Siewert and Kucerik, 2015). This is in accordance to Fierer et al. (2009), who argued that many soils share a common set of ecological mechanisms that govern biogeochemical processes. However, the connection of the fractions A–F to biogeochemical cycles has not been clarified yet, in particular C, D, and E which correspond to decomposition of SOM. We assume that if there is a connection, then they represent functional soil fractions and not only mathematical “artefacts”.

Therefore, the aim of this work is to answer the question about the meaning and potential functioning of the fractions, especially C, D and E. We assume that their potential function would be connected to content and dynamics of soil properties such as content of organic C, N, clay and microbiological stability. As aforementioned, the soil parameters can be obtained from TML reported in Table 1, thus we will perform the correlation analysis between TML (Table 1) and temperature areas (Table 2). The second aim is to validate the obtained

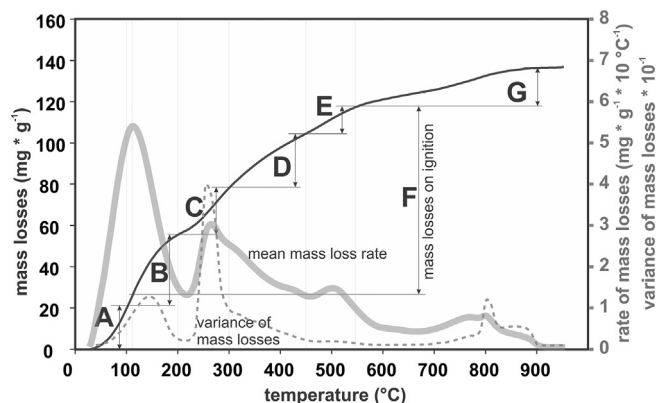


Fig. 1. An averaged TG and derivative TG (DTG) profile of soils investigated in this work and alternative way of fractionation (A–G).

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