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Assessing the effects of grinding on soil's thermogravimetric records: implications for carbon, nitrogen, and soil structure analysis

Eliška Kameníková[1] · **Ragačová Lucia**[1] · **M. Scott Demyan**[2] · **David Tokarski**[3] · **Martin Brtnický**[4] · **Jiří Holátko**[4] · **Jiří Kučerík**[4]

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Abstract

Soil structure is a key factor influencing its ecosystem functions. Previous research has shown a close correlation between mass losses obtained using thermogravimetry (TG) and soil properties such as the content of organic carbon and nitrogen, which has consequently enabled the determination of several relationships among obtained mass losses. The high degree of correlation has been explained, among other factors, as a result of the intact structure of the investigated soils. However, this hypothesis has never been experimentally tested. Therefore, this study investigates the effects of mild grinding, which primarily affects soil particles larger than 250 µm, on soil's TG records to determine its impact on the analysis of carbon, nitrogen, and relationship between mass losses. Soil samples from the island of Santorini, contaminated by heavy metals and dust from traffic, were analyzed with and without grinding using TG. Grinding affected the TG records across the entire temperature range, with the most significant decrease observed below 200 °C, where moisture evaporates. A mild increase was observed in the temperature range, where soil organic matter degrades. The determination of soil carbon and nitrogen content was only slightly impacted, which was explained as a result of only a small impact of grinding on soil microaggregates and organo-clay complexes. Despite these minor changes, as revealed by autocorrelation analysis, grinding significantly affected the relationships between mass losses. We conclude that soil grinding in TG analysis can be recommended for basic soil parameter analysis or contaminated soils due to improved homogeneity. However, it may compromise advanced analyses due to shifts in correlations between mass losses corresponding to the relationships between particular soil components.

Keywords Soil disruption · Thermogravimetry · Organic carbon · Nitrogen

Introduction

Soils provide a range of ecosystem services that are beneficial to the environment, human health, and the economy [1]. Among these services, carbon sequestration is particularly critical, as soil organic matter plays a central role in virtually all ecosystem functions. The other functions include supporting plant growth, offering habitats for biodiversity, water regulation and filtration, facilitating nutrient cycling, and regulating climate [2]. It is important to note that soil health, reflected by unaffected soil structure, is pivotal in these processes [3].

The structure and composition of mineral soils develop in response to ecosystem succession, starting from the weathering of parental rock, the accumulation of organic matter by and from soil biota, and the development of soil structure [4]. This includes the formation of the fundamental units of soil structure that are organo-mineral complexes and soil

✉ Jiří Kučerík
jiri.kucerik@mendelu.cz

[1] Institute of Chemistry and Technology of Environmental Protection, Faculty of Chemistry, Brno University of Technology, Purkyňova 118, 61200 Brno, Czech Republic

[2] School of Environment and Natural Resources, The Ohio State University, 2021 Coffey Road, Columbus, OH 43201, USA

[3] Faculty of Landscape Management, University of Applied Sciences Dresden, Pillnitzer Platz 2, 01326 Dresden, Germany

[4] Department of Agrochemistry, Soil Science, Microbiology and Plant Nutrition, Faculty of AgriSciences, Mendel University in Brno, Zemedelska 1, 61300 Brno, Czech Republic

aggregates, which co-exist in a certain equilibrium with labile fractions of soil organic matter (SOM). Organo-mineral complexes represent one of the main ways of stabilizing organic matter in soil. It is estimated that in cultivated soils, in temperate climate, more than 50% of soil organic matter occurs in organo-clay complexes [5]. Interaction of organic matter with mineral surfaces decreases the availability of carbon to soil microorganisms and increases its stability [5]. By binding together soil particles, these complexes contribute to the formation of soil aggregates. Soil aggregates are clusters of soil particles of variable size formed by the physical and chemical interaction of soil particles including living and non-living organic matter and minerals [6]. The formation and stability of soil aggregates are crucial because they support aeration and water infiltration (the space between aggregates), retention and capillarity (within macroaggregates), prevent soil erosion, and support biodiversity [7]. Soil aggregates contribute to carbon sequestration through several mechanisms, effectively trapping carbon and reducing its availability for microbial decomposition. The mechanisms include the physical protection of organic matter, the creation of a microenvironment inside the aggregates which is less favorable for microbial activity, and chemical stabilization by the formation of organo-mineral complexes inside the aggregate [8]. Importantly, organic molecules of SOM can change their occurrence and chemical and physical configuration several times during processes of aggregation and re-aggregation (taking weeks to months [9]) before they are mineralized [7].

Therefore, soil is an intricate ecosystem consisting of solid, liquid, and gaseous phases that integrates physical, chemical, and biological processes, functioning collectively as a unit [10]. This complexity is due to the diverse biological, organic, and inorganic substances it contains, which interact in ways that make soil arguably the most complex biological substrate on Earth [11]. Therefore, for its analysis, it is needed to employ a wide range of techniques. Recently, it has also been shown that it is advantageous to calibrate one technique to predict other properties. They include, among others, spectroscopic techniques such as FTIR [12], NIR [13], or the method of thermal analysis, thermogravimetry (TG) [14].

With the development of methods for analyzing the complex substrate that soil is, comes the question of proper sampling, sample preparation, and conditions of the employed measurement. Usually, sieving soil through a 2 mm sieve is chosen to separate undecomposed parts of fresh biomass such as roots, straw etc. and stones, which for most soil analyses is used as the cutoff for what is considered soil vs. coarse fragment, e.g. rock. Further reduction in size from the < 2 mm via grinding is sometimes used, which releases molecules and homogenizes the sample. This often has a positive influence on the quantitative analysis of certain components, especially when using spectroscopic techniques [15] and the reproducibility of TG records (personal experience). However, according to some authors [16], grinding soil can affect or erase relationships between individual components. In other words, grinding disrupts aggregates, organo-mineral complexes, pores, capillaries, releases molecules that were microbiologically and chemically stabilized, and changes the specific surface. This can especially affect TG analysis, which, if performed in an air atmosphere, is fundamentally based on the interaction of the atmosphere with the sample at higher temperatures. Therefore, molecules that would normally be protected from thermo-oxidation oxidize more easily at lower temperatures.

In this work, we focus on TG, which provides information about the mass of the sample exposed to a controlled heating environment. The mass losses in certain temperature ranges can be used to determine bound water content [17], the shares of labile and variously stabilized organic matter [18–20], the total content of soil organic matter [20] and the content of carbonates [21]. Furthermore, some temperature ranges are diagnostic for total nitrogen (TN) and soil organic carbon (SOC), clay content [21], and indicators of microbiological activity with respect to the amount of CO_2 released by soil microbes [22]. For soils preconditioned to atmosphere of 76% relative humidity (RH), mass loss between 340 and 350 °C or 320 and 330 °C can be used to determine SOC, while mass loss between 400 and 410 °C can be used to determine total organic nitrogen (TON) [21]. Some temperature ranges can then be used to determine additional microbial characteristics [23]. Furthermore, it has been shown that some mass losses in natural soils correlate with each other [16], thus providing the opportunity to analyze potential deficiencies in organic matter content [24]. They also allow for the analysis of various contaminations [25–27] and assist in evaluating the agronomic quality of soil [28, 29]. Some authors also use various indices, for example, TG-T50 refers to the temperatures at which half of the mass is lost during TG analysis [30].

There is not currently a consensus on a standardized samples preparation approach to grinding (personal communication), and it is often not clear in publications whether samples were finely ground prior to analyses, even from the methodological articles, e.g. [14, 31]. From the above-discussion, however, the question arises whether grinding could not help improve the sensitivity of TG for determining certain soil characteristics (or conversely reduce it). Some studies have suggested that specific conditions of measurement which preserve natural soil structure, i.e. unground soil (e.g. < 2 mm sieved) and equilibrated at a standard relative humidity, were the reason for the strong correlations between TML and C or N contents in natural soils unaffected by anthropogenic activities [32]. However, the assumption about the role of grinding has not been systematically investigated, therefore, research is needed to verify this hypothesis. Therefore, the aim of this work is to clarify whether grinding soils.

i. Significantly affects the TG record
ii. Affects the ability to use TG to determine C and N contents relative to unground soils
iii. Affects the mutual relationships between TMLs, representing soil characteristics

Furthermore, in this work, we tested soils sampled on the island of Santorini in 2018 for their properties with and without grinding by using thermogravimetry (TG). This locality was selected because it contains urban soils heavily contaminated by tourisms, mainly by traffic, which causes emissions of dust and acid rain, but does not affect soils mechanically. According to the Santorini airport information, on average, there were about 35 arriving flights per day. This translates to approximately 12,775 flights arriving at the airport over the course of the year 2018. In our previous works were tested mainly natural and agricultural soils, which properties variably, but still significantly, correlated with TG data. Therefore, including polluted soils can also clarify iv) whether the TG method is useful to determine fundamental characteristics also in contaminated soils.

Experimental

Soil sampling

Soils were sampled across four categories including airport, airport to ferry port road, urban roads, and rural roads, which are in details described in reference [33]. Samples were taken within 1 m from the edge of the road. Individual samples were a composite of five subsamples of the 0–10 cm soil depth, from an area of 1 m^2. After sampling, the samples were air-dried for two weeks and sieved to <2 mm, which was used for further analysis. In case of occurrence of small pieces of biomass residues such as straw or wood, they were mechanically removed. In total, 26 soil samples were analyzed for thermogravimetry. The samples were prepared in two different ways:

1. The air-dried, <2 mm samples (unground) were transferred to Petri dishes and placed in a desiccator. The desiccator contained two dishes of a supersaturated solution of $Ca(NO_3)_2$ to maintain an equilibrium relative humidity of 55% at 20 °C to ensure the same moisture conditions during TG measurements, in the same way as in previous works e.g. Ref. [34]. Samples were left for three weeks before measurement to ensure constant relative humidity.
2. The second method of sample preparation was like the first procedure, except samples were finely ground prior to being placed in the desiccator. The samples were ground in a Retsch MM200 laboratory ball mill for one minute at 25 Hz in a stainless steel grinding vessel with a volume of 25 mL (grinding vessel was filled halfway). Two stainless steel balls were used during the milling process. These parameters represent the shortest time providing visually homogeneous ground soils, while keeping soil microaggregates intact. The ground samples were then equilibrated at 55% relative humidity for three weeks the same as the unground samples.

Soil characteristics

Soils were analyzed for carbon and nitrogen content by an elemental analyzer. The ground samples were dried to constant mass at 105 °C, and the total carbon content was determined according to ISO 10694: 1995 and the total nitrogen content was determined according to ISO 13878: 1998 both on a LECO TruSpec analyzer (MI, USA). The averaged results are shown in Table S1 (SI).

In order to observe the effect of grinding, the air-dried soils were subjected to the particle size analysis. Both ground and unground soils were analyzed using sieves with screen diameters 1 mm, and 500, 250, 125, and 63 μm. The fractions were weighed by using laboratory analytical scales.

Thermogravimetry (TG) of soils

The TG analysis of ground and unground soils was conducted using a TGA 550 (TA Instruments, Delaware, USA). The TG analyzer, both the sample feeder and oven, were preconditioned to maintain conditions at 55% relative humidity. As part of the preconditioning, a plastic cover was created for the sample feeder, from which three tubes lead, ensuring the supply of air with the necessary humidity. This allowed to maintain the required air humidity at the feeder. An aquarium pump was used for air pumping, and to enrich the air to 55% humidity, a system of three wash bottles interconnected by rubber tubes was used. In the first wash bottle, water was placed; in the second, a supersaturated solution of $Ca(NO_3)_2$, (prepared by dissolving/suspending 1.5 kg of the salt in one liter of water) which is capable of capturing excess air humidity brought from the wash bottle with water and adjusting it to the air humidity value of 55%. In the third bottle, cotton wool was placed to catch any small crystals of $Ca(NO_3)_2$ that could enter the system and potentially clog it. This three-bottle system was connected to the tubing from the gas source and thus connected to the TG furnace and sample reservoir. The soil samples (typically 150–200 mg) were heated at 5 °C min^{-1} under dynamic atmosphere of air enriched to 55% RH (at 20 °C), flow rate 100 mL min^{-1}, pan type Al_2O_3 from laboratory temperature (typically 25 °C) to 700 °C. Mass change was recorded once per six seconds in order to reduce number of points for further analysis.

Each sample was measured at least in triplicate. An arithmetic mean from all measurements was used for the purposes of evaluation. The mass loss data were across 10 °C intervals as described in earlier works, e.g. Ref. [21]. The 10 °C mass losses were used for further analysis. For example, we express mass loss between e.g. 50 and 60 °C as thermal mass loss TML_{60}.; mass loss between e.g. 300 and 450 °C is expressed as $\mathrm{LTML}_{300\text{-}450}$ (large TML).

For the data analyses, Excel and R Studio software were used. In particular, the correlation analyses were conducted using Excel, the autocorrelation was calculated in Excel, visualized using Origin. The R Studio (own syntax) was used to extract the data to create the distribution histograms. Origin also served for visualization of other data. Notably, for the 26 samples investigated in this study, statistical significance at the 5, 1, and 0.1% levels corresponds to linear correlation coefficients (r) of approximately 0.39, 0.51, and 0.62, respectively.

To verify the effect on the quantifiability of organic C and total N (TN) in ground soils, we used equations from the previous work of Kučerík et al. [34]. For the calculation of TN, the Eqs. 1–3 were used.

$$\mathrm{TN} = 0.09 \times \mathrm{TML}_{290} + 0.03 \quad (1)$$

$$\mathrm{TN} = 0.14 \times \mathrm{TML}_{330} + 0.02 \quad (2)$$

$$\mathrm{TN} = 0.19 \times \mathrm{TML}_{410} + 0.02 \quad (3)$$

For the calculation of SOC were used Eqs. 4 and 5

$$\mathrm{SOC} = 1.18 \times \mathrm{TML}_{330} + 0.17 \quad (4)$$

$$\mathrm{SOC} = 1.33 \times \mathrm{TML}_{350} + 0.22 \quad (5)$$

Equations for the calculation of fractions were also tested, namely Eqs. 6–8.

$$\mathrm{LTML}_{110-550} = 1.42 \times \mathrm{TML}_{80} + 24.1 \times \mathrm{TML}_{330} + 1.51 \quad (6)$$

$$\mathrm{LTML}_{110-550} = 2.49 \times \mathrm{TML}_{80} + 14.6 \times \mathrm{TML}_{290} + 1.4 \quad (7)$$

$$\mathrm{LTML}_{30-550} = 7.18 \times \mathrm{TML}_{80} + 15.9 \times \mathrm{TML}_{290} + 1.9 \quad (8)$$

The TML values obtained for ground soils (right side of the equations) were used, and the calculated LTML was compared to the LTML measured using TG. The results were evaluated based on the percentage deviations.

Results

Comparison of particle sizes

Figure 1 reports averaged results ($n = 26$) of relative mass fraction of particle sizes for both ground and unground soils within the size intervals 2–1 mm, 1 mm–500 µm, 500–250 µm, 250–125 µm, 125–63 µm and fractions below 63 µm. The data indicate that grinding resulted in a reduction in particle mass fractions in the larger size intervals, with particles shifting to smaller sizes. The switch from the relative fractions´ abundances occurred at 250 µm.

As shown in Fig. 1, the relative mass fractions of smaller-sized fractions increased in soils that were ground.

Comparison of TG records

To evaluate the TG of both sets of samples, TMLs from both ground and unground soils were used, the values were averaged and plotted in graphs. Figure 2 shows this comparison, and it is evident that the largest difference occurred primarily in the area up to 200 °C, where TML were higher for unground soils. Other differences were also noticeable in the area from 300 to 350 °C and then also in the area from 400 to 500 °C, where TML were higher for ground soils.

The impact of grinding on the determinability of carbon and nitrogen

In this section, we focus on comparing the effects of soil grinding on the ability to determine total carbon and nitrogen content, as illustrated in previous studies [34]. The

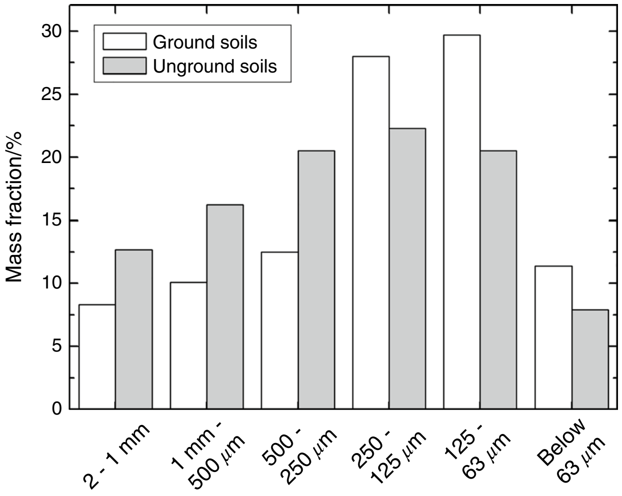

Fig. 1 Comparison of averaged mass fractions of particle sizes of ground and unground soils ($n = 26$)

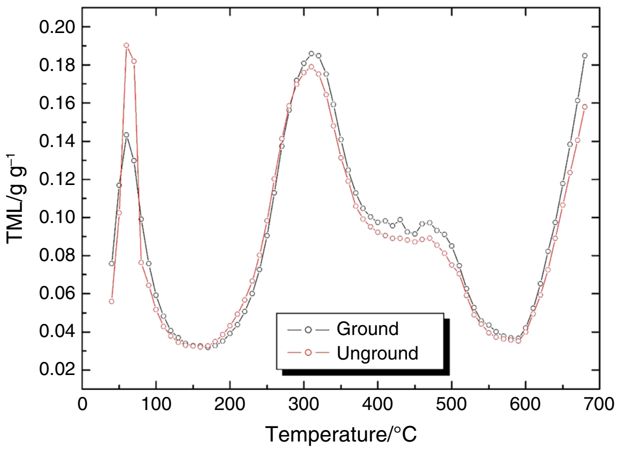

Fig. 2 Comparison of averaged TG records of ground and unground soils ($n=26$)

comparison was carried out by correlating the results from elemental analysis with TML. Figure 3 presents the correlation coefficient r between the total carbon content and TML data across temperatures for both ground and unground soil samples in order to observe the differences caused by grinding and possible shifts in temperatures with highest correlations. Generally, the correlations were slightly higher for unground soils across most temperature ranges. The findings shown in Fig. 3 indicate that the highest correlation for total carbon content was 0.87 for unground soils and 0.84 for ground soils at TML_{490}. In the temperature range of 300 to 350 °C, which was identified as having the highest correlation in previous studies [21, 34], the coefficients were below 0.8 for both ground and unground soils.

The correlation between total nitrogen content in both ground and unground soil samples, in relation to the correlation coefficient r and temperature, is shown in Fig. 4. In this instance as well, correlations were marginally higher for unground samples. Notably, higher correlation coefficients were recorded in the temperature range of 430–510 °C, with the peak correlation at TML_{450} for both sample types, where the correlation reached 0.97.

The comparison of calculations showed that grinding slightly worsens the quantifiability of TN and SOC defined by equations of 1–3 and 4–5, respectively. The comparison of ground soil with the equations exhibited only small differences, negligible for regular practice. For the SOC determination the difference was by a maximum of 0.8% and for TN by a maximum of 2.1%.

Additionally, equations for determination of larger mass fractions depicted in Eqs. 6–8 exhibited only slightly worse results, with calculated differences of approximately 1.9, 6.9, and 1.5%, respectively.

Autocorrelation of TG data

Figures 5 and 6 display the raster autocorrelation matrixes (heat map) of TML_{30} to TML_{650} in the temperature interval 30–650 °C (the interval relevant to observe moisture, SOM and carbonates). That means that the plot consists of $63 \times 63 = 3969$ raster points. Each pixel in the Figures represents a coefficient of determination (R^2) between corresponding TMLs ($n=26$), which is shown as a white–gray-black scale and corresponds to the correlation between TMLs obtained at temperatures displayed on the X and Y axes. The darker the pixels, the higher were the correlations between TML in the corresponding 10 °C temperature intervals. A diagonal white line from the top left corner to the bottom right corner represents correlations of TML in the same temperature intervals ($R^2=1$), thus, this line is

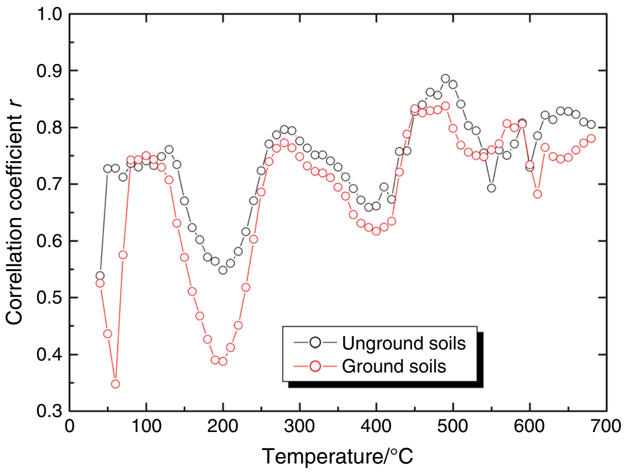

Fig. 3 Correlation coefficients between SOC and TML for both ground and unground soils ($n=26$)

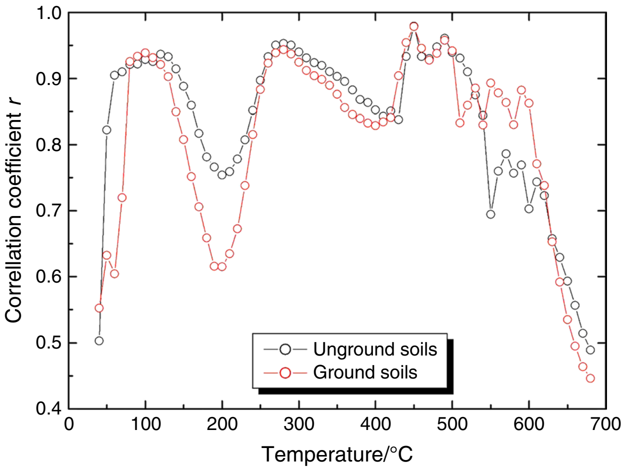

Fig. 4 Correlation coefficients between total nitrogen and TML for both ground and unground soils ($n=26$)

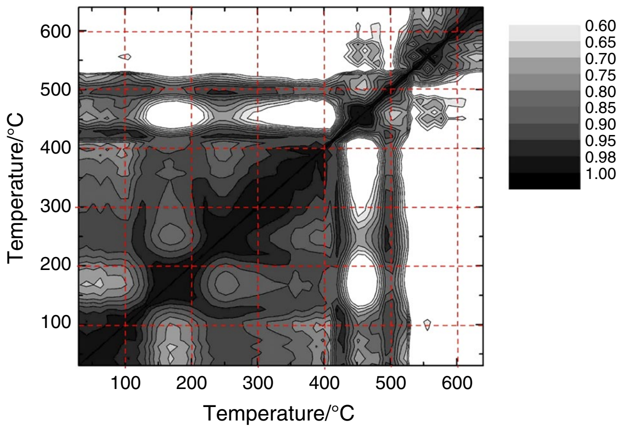

Fig. 5 A heat map of coefficients of determination (R^2) between individual TMLs for unground soils

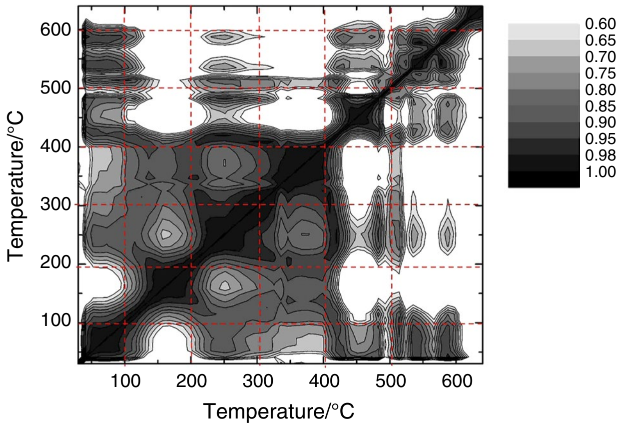

Fig. 6 A heat map of coefficients of determination (R^2) between individual TMLs for ground soils

irrelevant. However, separated gray and black intervals along the diagonal line allow for the grouping of soil components that are characterized by narrower autocorrelations [32].

For unground soils (Fig. 5), mutual correlations occurred in the temperature ranges of 25–170, 170–250, 250–450, 450–550, and 550–680 °C. There was also a strong correlation between the ranges of 450–460 and 25–150 °C as well as 250–380 °C. Additionally, the 510–530 °C range correlated with 30–150 and 220–380 °C. Conversely, for ground soils (Fig. 6), correlations were observed in narrower intervals: 80–150, 170–240, 250–450, and 450–500 °C. The correlation between the 450–460 °C range and the 25–150 °C range was significantly weaker, while the correlation with the 250–380 °C range was as strong as in the case of unground soils. The 550–610 °C range also correlated with 30–150 °C.

To facilitate the comparison of autocorrelation matrices, Figures S1 and S2 (SI) display histograms of correlation coefficient frequencies across both records in the total range of 30–650 °C. It is evident that unground soils had a higher frequency of higher correlation coefficients, i.e. in the interval 0.95–1 (14.1%), 0.9–0.95 (14.6%) and 0.85–0.9 (10.4%) compared to ground soils, which showed mass fractions in intervals of 0.95–1 (9.8%), 0.9–0.95 (8.1%) and 0.85–0.9 (9.2%).

Discussion

Influence of grinding on water binding

As expected, even mild grinding soil caused small shifts in the particle distribution (Fig. 1) and affected the TG records across most temperature ranges (Fig. 2). In the 30–200 °C range, a decrease in mass loss was observed for the ground sample, which can be attributed to a change in moisture content caused by the disruption of the original soil structure. In other words, under the same conditions of relative humidity, to which the samples were exposed before analysis, less water was bound to the resulting particles. As mentioned in the introduction, soil structure is complex and developed during pedogenic processes, containing a range of pores and cavities, where not only physical sorption of water on the soil surface occurs, but also condensation and formation of water clusters [35].

The soil structure also inherently includes a variety of interactions between different soil components [10, 16]. Grinding disrupted these relationships, as shown by the autocorrelation analysis (Fig. 6), although, surprisingly, some relationships remained intact. It remains a question whether changing the grinding conditions, such as extending the time, would disrupt these relationships even more.

The soils tested in this work showed several correlations between distant temperature ranges such as 450–460 and 25–150 and 250–380 °C. For natural soils exposed to 76% RH, Siewert [21] observed a correlation between TMLs 530 and 120 °C, which was interpreted as a correlation between diagnostic temperatures for formation of soil organo-clay complexes [32] (RH shifts the diagnostic temperatures for clays [34]). This corresponds with the first mentioned correlation, while the second correlation had not been observed before. Surprisingly, these correlations remained preserved for ground soils, albeit with reduced intensity. We can only speculate that this is due to an effect observable only in these soil samples.

Influence of grinding on soil aggregates

Further differences in the TG records were observed in the range from 300 to 450 °C, where higher mass changes were noted for ground soils. This area is associated with the degradation of both labile and organically stabilized matter within aggregates [18]. Soil aggregates consist of clay, silt, and sand particles containing various cations (e.g., Ca^{2+}) bound together by organic matter. This includes decaying plant and animal residues, soil organisms such as bacteria, fungi, and earthworms, and residual stable organic matter and substances produced by these organisms, like polysaccharides and glomalin. These substances act as natural glue, binding soil particles together. Mechanically, macroaggregates (> 250 µm) are less stable than microaggregates (< 250 µm), which are considered to be very stable even mechanically [36]. Figure 1 indicates that content of macroaggregates decreased due to the grinding. The results thus suggest that grinding likely led to the release of molecules from the macroaggregates compared to unground samples (Fig. 2), where molecules remained partially protected. According to Blanco-Canqui, and Lal [37], the macroaggregates are formed and stabilized by the C-rich young plant residues, while the microaggregates occlude mainly the old organic C. This aligns with the observation that plant residues degrade above 300 °C [24] and in this work contributed to the mass loss increase in the temperature area 300–450 °C.

Importantly, destroying the macroaggregates by grinding also negatively influenced the water binding; the released organic matter and residual microaggregates were not effective in water holding as in the macroaggregates, where water is trapped in pores and capillaries and thereby significantly contributing to water retention in soils [38].

Grinding also significantly changed the autocorrelation of TMLs in the temperature range from 300 to 450 °C, indicating a change in the relationships between components thermally degraded in this area. This area is also typically designated as a diagnostic area for determining soil organic carbon in natural soils. In this work, the highest coefficients were observed at TML_{490}, although TML_{330} also shows a high correlation. Even though grinding changed the mass losses, and increased the mass of ground soils in this area, the correlation coefficient between TML and SOC decreased only slightly. The shift in diagnostic temperature may be caused by several factors. As mentioned, one of the factors could be soil moisture, which affects the temperature of maximum correlation [34]. Contamination may play an important role, as the soils originate from areas heavily affected by traffic leading to the elevated content of heavy metals [33] and carbonaceous particulate matter [39]. Furthermore, soils originate from volcanic parent materials [33], therefore, they contain a higher content of natural pyrogenic carbon influencing diagnostic temperatures [32].

Influence of grinding on organo-mineral complexes

Further changes in the TG records were observed in the range of 450 to 600 °C, where TML was higher for ground soils. In this area, organo-clay complexes are degraded, hence, it can be presumed that they are also partially affected by grinding (although this could not be observed in the particle size distribution in Fig. 1). SOM interacts with clay minerals through various mechanisms, including hydrogen bonding, electrostatic interactions, van der Waals forces, and ligand exchange. Grinding can significantly affect organo-clay complexes by altering their physical and chemical properties [40]. The extent of these effects largely depends on the milling method, intensity, duration, and the specific characteristics of both the organic matter and the clay minerals involved [41]. Grinding can reduce the particle size of clay minerals, increase their specific surface area, lead to the delamination of layered clays, or even change their crystal structure. This might either enhance or reduce the stability of organo-clay complexes. The mechanical forces during grinding create reactive sites on the clay surface, facilitating chemical interactions with organic molecules, potentially forming new organo-clay complexes with different characteristics from those formed without milling [40]. Additionally, some grinding processes generate significant heat, which could cause thermal degradation of organic molecules or alter the thermal properties of the clay, affecting the nature and stability of the organo-clay complexes formed.

The 400 to 500 °C range is diagnostic for TN, with TML_{420} for 76% RH [21] while TML_{290} correaltes at 43% RH [34]. In this study, the highest correlation was observed at TML_{450}, and although the correlation coefficient was above 0.8 across the 250–450 °C range, grinding did not affect the correlation coefficient. As already discussed, shifts in diagnostic temperature may be caused by various factors such as contamination, soil origin, or the RH used during soil conditioning before measurement.

Conclusions

In this work we aimed to answer the questions whether the grinding significantly impacts TG results, affects the ability to use TG to determine C and N and affects the mutual relationships between TMLs representing soil characteristics and whether TG can be used for analysis of soil contaminated by heavy metals from traffic. The results showed that even mild grinding impacts the TG record over whole temperature range, the most intensively up to 200 °C, which is mainly loss of soil moisture. Despite the changes in the soil structure, mild grinding did not significantly affect the determination of soil C and N contents, the results showed a close correlation between mass losses and those two parameters. On the contrary, mild grinding of soil had an impact on the mutual relationships between soil components represented by mass loss in specific temperature areas. Therefore, for specific analyses, in which only basic soil parameters are determined (except for soil moisture content) or for soils containing particulate contaminants, grinding can be recommended as it provides better soil homogeneity. Nevertheless, for advanced soil analysis, grinding shifts some correlations between soil components, which can compromise obtained results.

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Author's contribution EK was involved in controlling the TG experiments, particle size analysis, data evaluation, and writing; LR was involved in analyses, and partial data analysis; MSD was involved in methodology, and writing; DT was involved in data analysis, and writing; MB was involved in soil sampling, soil analyses, and writing; JH was involved in soil analyses, statistical analysis, and methodology; JK was involved in supervising, writing, data analysis and conceptualization**.** All authors read and approved the final manuscript.

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