



Article

The Effect of Heating on Properties of Sandy Soils

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Abstract: Although burning grass and crop residues is prohibited in many countries, farmers perceive it as a quick and inexpensive way to eliminate unwanted biomass. The aim of this study was to estimate the impact of heating temperature (simulation of biomass burning) on the studied properties (soil organic carbon (SOC) content, pH(H₂O), water drop penetration time, WDPT, and contact angle, CA) of acidic sandy soils. Soil samples were taken from the experimental sites S1, S2, and S3 at Studienka village in the Borská nížina lowland (southwestern Slovakia). Experimental site S1 was arable land, experimental site S2 was arable land abandoned for approximately 10 years, and experimental site S3 was arable land abandoned for approximately 30 years with scattered Scots pine (Pinus sylvestris L.) trees. It was found that all the soil properties studied were strongly affected by heating. A drop in SOC was observed in all the soils for the heating temperature between 20 and 600 °C. Due to the incomplete combustion of SOC, a small (0.1–0.7%) SOC content was recorded even in soils heated to between 600 and 900 °C. An increase in pH(H₂O) was observed in all the soils for the heating temperature higher than 300 °C. Soil from the experimental site S1 was wettable (WDPT < 5 s) for all of the heating temperatures. WDPT vs. heating temperature relationships for the soils from the experimental sites S2 and S3 were more complex. After a decrease in the heating temperature of 50 $^{\circ}$ C, an increase in WDPT for the heating temperature between 50 $^{\circ}$ C and 300 $^{\circ}$ C (for S3 soil) and 350 °C (for S2 soil) was registered. Finally, the WDPT dramatically dropped to 0 for the heating temperature of 350 °C (for S3 soil) and 400 °C (for S2 soil). CA started to decrease at 300 °C in all the soils and dropped to 0° for all the soils at 800 °C. CA > 0° measured in soils for the heating temperature between 400 and 800 °C, as a consequence of the small SOC contents due to the incomplete combustion of SOC, is a novelty of this study which demonstrates that CA is more sensitive to the changes in subcritical water repellency than WDPT.

Keywords: sandy soil; soil water repellency; soil heating; soil organic carbon; pH; water drop penetration time; contact angle

1. Introduction

Although burning grass and crop residues is prohibited in many countries, farmers perceive it as a quick and inexpensive way to eliminate unwanted biomass. Farmers historically used straw burning in the Central European region to eliminate weeds, pests, and excess biomass while fertilizing soils [1]. Farmers believe burning crop residue is crucial in controlling weeds and pests through direct destruction or alteration of their natural habitat. They also believe that potassium is gained from the ash for the next crop cycle. Although this may be true in the short term, the consequences of losing other essential nutrients and organic carbon outweigh the short-term benefits [2]. Rasoulzadeh et al. [3] found that burning crop residues may reduce saturated hydraulic conductivity, sorptivity, and infiltration rate.



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Systematic fire suppression and exclusion became routine in the 20th century. In the previous decades, the negative consequences of systematic fire exclusion have been shown for grassland ecosystems. Armas-Herrera et al. [4] showed that prescribed grassland burning is a tool to reduce hazardous fuels, improve foraging for grazing, improve wildlife habitat, manage competing vegetation, perpetuate fire-dependent species, control insects and disease, and cycle nutrients. The role of plant species adapted to fire events on runoff and soil erosion was studied by Cerdà et al. [5]. Bonanomi et al. [6] found that burning and mowing a Mediterranean grassland significantly increased plant species richness and diversity compared to abandoned plots, by reducing the abundance of perennial tall grasses in favor of short-lived species. Compared with abandoned plots, prescribed burning increased soil pH and reduced the stock of organic carbon, total nitrogen, total carbonates, and cation exchange capacity. Tulganyam and Carr [7] found that the burned and unburned treatments concerning perennial grass production were similar. The fire line treatment appeared to trigger an exotic annual grass invasion.

Laboratory heating of soil samples in muffle furnaces is often used to simulate a natural fire. However, muffle furnaces heat the samples from all sides, making them significantly different from natural fires that only heat the soil surface [8]. Novák et al. [9] found that water drop penetration time (WDPT) and saturated hydraulic conductivity of sandy soils from grassland increased with increasing the heating temperature from 20 to 250 °C. Stoof et al. [10] revealed that heating soil to <200 °C did not change the properties of the studied sandy soil, but heating soil to >300 °C increased bulk density, clay and silt content, and decreased soil organic matter and sand content. Hološ et al. [11] revealed that the fire-induced changes in soil properties depend on the age and type of forests. Martínez et al. [12] found that pH increased with the heating temperature to 300–500 °C and then decreased to 900 °C. The saturated and unsaturated (negative tension of -3 cm) hydraulic conductivity of sandy loam soil increased, while soil organic matter content decreased with the heating temperature.

Agricultural land abandonment, often called the cessation of farming and giving away of land for natural succession, is widespread in many regions, including Europe. Passive revegetation in permanently abandoned arable land (secondary succession) is characterized by the replacement of arable plant species by vegetation that disperses from surrounding habitats and will be subsequently established [13]. Similar to studies by Hewelke [14] and Hewelke et al. [15] in sandy soils, our previous studies also found that soil organic carbon (SOC) content, repellency index (RI), and WDPT increased and pH decreased in acidic sandy soil in Studienka, southwestern Slovakia, and alkaline sandy soil in Csólyospálos, south Hungary, during their 30- and 44-year abandonment, respectively [16,17]. Soil water repellency induced by the vegetation change during succession and parameterized by WDPT and contact angle, CA, is influenced by soil moisture, texture, pH, SOC, CaCO₃, and clay (mainly kaolinite) content [18–20].

The aim of this study was to estimate the impact of heating temperature on the selected properties (SOC, pH(H_2O), WDPT, and CA) of acidic sandy soils taken from the experimental sites selected to include three different stages of secondary succession on abandoned fields. Based on the results of our previous study on the heating of sandy soil [11], we hypothesized that: 1. SOC will decrease with the heating temperature in the interval 20–900 °C; 2. pH will decrease with the heating temperature in the interval 20–300 °C and then increase to 900 °C; 3. WDPT will dramatically drop to 0 for the heating temperature of 350–400 °C; and 4. CA will decrease to 0° for the heating temperature of 350–400 °C.

2. Material and Methods

2.1. Site Description

Soil samples were taken from the experimental sites S1, S2, and S3 at Studienka village in the Borská nížina lowland (southwestern Slovakia) (Figure 1). The sites were selected to include three different stages of secondary succession in relatively same site condi-

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tions (climate, soil, and relief conditions). Experimental site S1 (48°31′37″ N, 17°7′54″ E; 173 m a.s.l.) is arable land with flat relief. It is covered by synanthropic vegetation dominated by annual herbs, replacing harvested crops. Experimental site S2 (48°31′41″ N, 17°7′49″ E; 180 m a.s.l.) is arable land abandoned for approximately 10 years with flat relief. It is covered by synanthropic vegetation dominated by perennial grasses. Experimental site S3 (48°31′39″ N, 17°7′52″ E; 171 m a.s.l.) is arable land abandoned for approximately 30 years with scattered Scots pine (*Pinus sylvestris* L.) trees with flat relief. It is covered by synanthropic vegetation dominated both by perennial grasses and perennial herbs. More detailed information about the vegetation composition on the experimental sites can be found in Toková et al. [17].

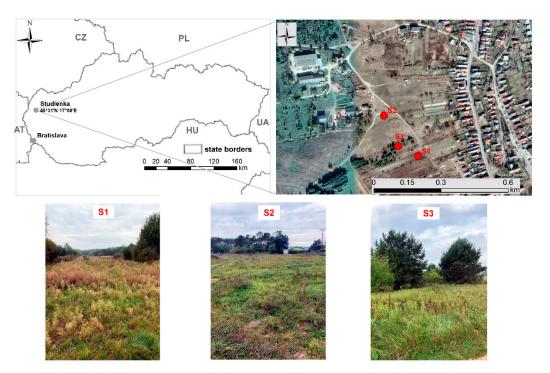


Figure 1. Location and aerial view of the studied area with experimental sites S1, S2, and S3 located at Studienka village in the Borská nížina Lowland (southwestern Slovakia).

Experimental sites are situated in a region with a temperate oceanic climate (Cfb) [21]. The mean annual temperature is 9 $^{\circ}$ C, the mean annual precipitation is 600 mm, with most rainfall occurring during the summer season. The soils of the Studienka sites are classified as Arenosol and have a sandy texture [22]. The basic physical and chemical properties of the sampled soils are presented in Table 1.

Table 1. Physical and chemical properties of the top (0-5 cm) soils taken from experimental sites S1, S2, and S3. The results are presented in the form: arithmetic mean \pm standard deviation (the number of repetitions = 2). (SOC—soil organic carbon).

Attribute	S1	S2	S3
Sand (%)	91.94 ± 0.54	95.38 ± 0.14	94.50 ± 0.03
Silt (%)	2.41 ± 0.43	1.57 ± 0.06	1.53 ± 0.05
Clay (%)	5.66 ± 0.12	3.06 ± 0.08	3.97 ± 0.02
CaCO ₃ (%)	< 0.05	< 0.05	< 0.05
SOC (%)	$0.99~^{\rm a} \pm 0.01$	$1.93^{\rm \ b} \pm 0.02$	$1.67^{\rm \ b} \pm 0.01$
pН	$6.25~^{\mathrm{a}}\pm0.01$	$6.78~^{\mathrm{a}}\pm0.01$	$6.19~^{\mathrm{a}}\pm0.01$

Properties denoted with different letters are significantly different on significance level 0.05.

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2.2. Soil Sampling and Heating Experiment

The 200 cm \times 200 cm area was marked out at each experimental site, and approximately 6 kg of soil was taken from its top layer (0–5 cm) and placed in plastic containers. The soil was then sifted through a 2 mm sieve in the laboratory, mixed, and air-dried at ambient temperature. After the drying process, 60 g of soil was placed in ceramic dishes, with five dishes allotted for each temperature. Using a muffle furnace (LAC, s.r.o., Židlochovice, Czech Republic; Type: LE 15/11), the dishes with soil were heated for 20 min at temperatures ranging from 50 to 900 °C (with an increment of 50 °C), when each sample was used for only one temperature. After heating, the samples were cooled to ambient temperature. A diagram showing the experimental design with the number of samples and their repetitions is presented in Figure 2.

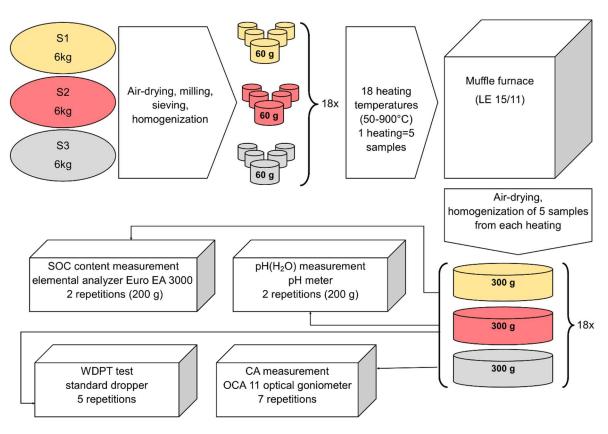


Figure 2. Diagram showing the experimental design with the number of samples and their repetitions.

2.3. Measurement of Soil Properties

Basic soil properties were determined on disturbed samples in the ISO-certified Laboratory of the Soil Science and Protection Research Institute in Bratislava. Particle size distribution was determined by sieving and sedimentation according to ISO 11277 [23] with 2 repetitions for each experimental site and heating temperature. The pH($\rm H_2O$) was measured according to ISO 10390 [24], with 2 repetitions for each experimental site and heating temperature. SOC content was determined in the elemental analyzer Euro EA 3000 (HEKAtech GmbH, Wegberg, Germany) with 2 repetitions for each experimental site and heating temperature.

The persistence of soil water repellency (SWR) was estimated by the water drop penetration time (WDPT) test, which involves placing $50 \pm 5~\mu L$ of a drop of water from a standard dropper or pipette on the soil surface and recording the time of its complete penetration (infiltration) into the soil. An average drop release height of approximately 10 mm above the soil surface was used to minimize the cratering effect on the soil surface [25,26]. The following classes of the persistence of SWR were then distinguished: wettable or non-water-repellent soil (WDPT < 5 s), slightly (5 s \leq WDPT < 60 s), strongly

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 $(60 \text{ s} \le \text{WDPT} < 600 \text{ s})$, severely $(600 \text{ s} \le \text{WDPT} < 3600 \text{ s})$, and extremely $(\text{WDPT} \ge 3600 \text{ s})$ water repellent soil [27]. WDPT was estimated with 5 repetitions for each experimental site and heating temperature.

The severity of SWR was estimated by the sessile drop method. This involved placing a water drop on the soil sample's surface and analyzing the static contact angle (CA) by reviewing image recordings taken with the optical goniometer OCA 11 (DataPhysics Instruments GmbH, Filderstadt, Germany). The procedure described by Bachmann et al. [28] was used to prepare the samples, which involved covering a glass slide with double-sided adhesive tape and pressing soil particles onto the tape for several seconds. The slide was shaken carefully to remove any unglued soil particles and then a 5 µL drop of deionized water was placed on the sample surface using a 0.91 mm syringe needle. After 1 s when mechanical disruption of the surface was complete after drop placement, CA was evaluated by analyzing the shape of the drop (ellipsoid approximation) and fitting tangents on both sides of the drop using dpiMAX version 1.51.90.75 software (DataPhysics Instruments GmbH, Filderstadt, Germany) according to Goebel et al. [29]. CA of each drop was determined as an arithmetic mean of the CA values on the left and right sides of the drop. The following classes of the severity of SWR can be distinguished: non-water-repellent soil (wettable) $(CA < 40^{\circ})$, slightly $(40^{\circ} \le CA < 90^{\circ})$, moderately $(90^{\circ} \le CA < 110^{\circ})$, strongly and very strongly ($110^{\circ} \le CA < 130^{\circ}$), and extremely ($CA \ge 130^{\circ}$) water repellent soil [30]. CA was estimated with 7 repetitions for each experimental site and heating temperature.

2.4. Statistical Analysis

To determine if the data follow a normal distribution, we utilized an omnibus test that considers both skewness and kurtosis. If the normality assumption is met, we proceeded with a single-factor ANOVA and Tukey's Honest Significant Difference (HSD) post-hoc test to compare various parameters. However, if the normality assumption is not met, we applied the Kruskal–Wallis test and its Z test for multiple comparisons. This test does not require normality assumptions and is specifically utilized to compare medians. Our threshold for statistical significance was set at a *p* value below 0.05, and all statistical analyses were performed using the NCSS 12 version 12.0.18 statistical software [31].

3. Results

The impact of heating temperature on SOC, pH, WDPT, and CA of sampled soils is presented in Figures 3–6. A drop in SOC was observed in all the soils for the heating temperature from 20 to 600 °C. Due to the incomplete combustion of SOC, the small (0.1–0.7%) SOC contents were recorded even in soils heated to 600–900 °C, which confirms our first hypothesis. Statistically significant differences in SOC (median of SOC values for temperatures 20–900 °C) were found for soils from 1- and 10-year abandoned fields and for soils from 1- and 30-year abandoned fields. A statistically significant difference in SOC between 10- and 30-year abandoned fields was not found (Figure 3).

An increase in pH(H₂O) was observed in all the soils for the heating temperature higher than 300 °C, which confirms our second hypothesis. A decrease in pH(H₂O) for the heating temperature 20–300 °C was observed only in S3 soil. Statistically significant differences in pH (median of pH values for temperatures 20–900 °C) for soils from 1- and 10-year abandoned fields and for soils from 10- and 30-year abandoned fields were observed. A statistically significant difference in pH between 1- and 30-year abandoned field was not found (Figure 4).

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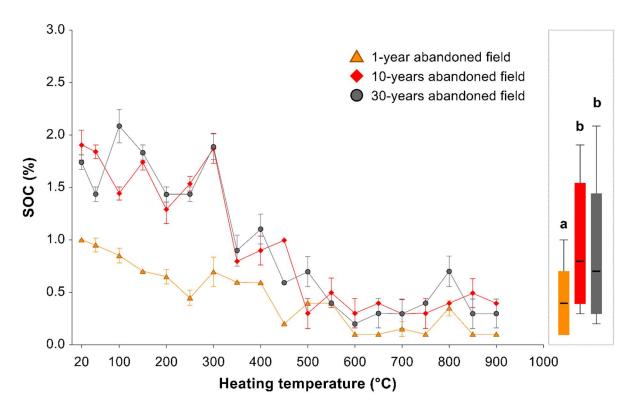


Figure 3. Soil organic carbon (SOC) content vs. heating temperature relationships and box plots for soil samples from 1-, 10-, and 30-years abandoned fields in Studienka, southwestern Slovakia. The results of SOC are presented in the form: arithmetic mean \pm standard deviation, when the number of repetitions was 2. The box plots denoted with different letters are significantly different on significance level 0.05.

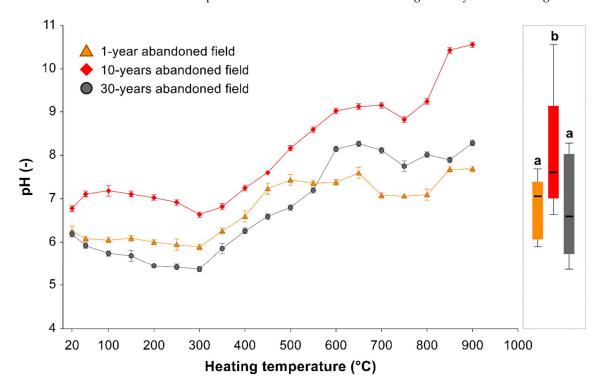


Figure 4. The pH(H₂O) vs. heating temperature relationships and box plots for soil samples from 1-, 10-, and 30-years abandoned fields in Studienka, southwestern Slovakia. The results of pH(H₂O) are presented in the form: arithmetic mean \pm standard deviation, when the number of repetitions was 2. The box plots denoted with different letters are significantly different on significance level 0.05.

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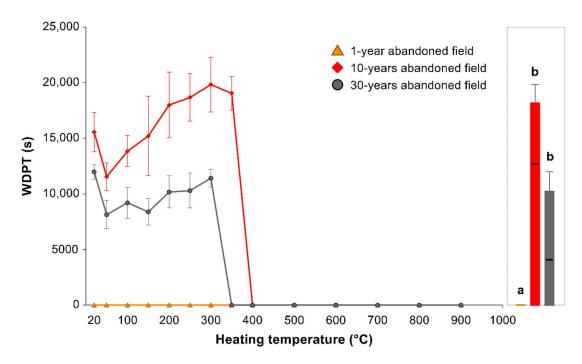


Figure 5. Water drop penetration time (WDPT) vs. heating temperature relationships and box plots for soil samples from 1-, 10-, and 30-years abandoned fields in Studienka, southwestern Slovakia. The results of WDPT are presented in the form: arithmetic mean \pm standard deviation, when the number of repetitions was 5. The box plots denoted with different letters are significantly different on significance level 0.05.

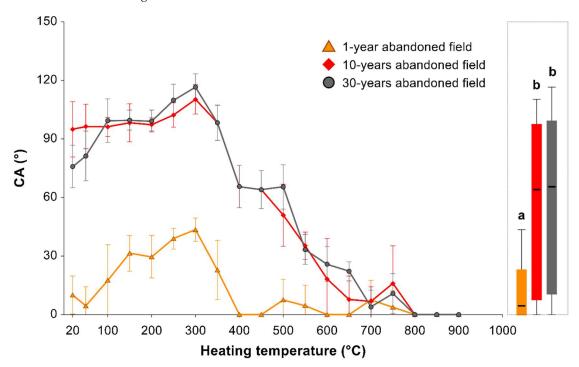


Figure 6. Static contact angle (CA) vs. heating temperature relationships for soil samples from 1-, 10-, and 30-years abandoned fields in Studienka, southwestern Slovakia. The results of CA are presented in the form: arithmetic mean \pm standard deviation, when the number of repetitions was 7. The box plots denoted with different letters are significantly different on significance level 0.05.

Soil from the experimental site S1 was wettable (WDPT < 5 s) for all of the heating temperatures. WDPT vs. heating temperature relationships for the soils from the experimental

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sites S2 and S3 were more complex. Initially, the WDPT of unheated soil decreased for the heating temperature of 50 $^{\circ}$ C; then, an increase in WDPT for the heating temperature from 50 $^{\circ}$ C to 300 $^{\circ}$ C (for S3 soil) and 350 $^{\circ}$ C (for S2 soil) was registered. Finally, the WDPT dramatically dropped to 0 for the heating temperature of 350 $^{\circ}$ C (for S3 soil) and 400 $^{\circ}$ C (for S2 soil), which confirms our third hypothesis. Statistically significant differences in WDPT (median of WDPT values for temperatures 20–500 $^{\circ}$ C) for soils from 1- and 10-year abandoned fields and for soils from 1- and 30-year abandoned fields were observed. A statistically significant difference in pH between 10- and 30-year abandoned fields was not found (Figure 5).

CA started to decrease at 300 °C in all the soils, dropped to 0° for the first time in S1 soil at 400 °C, and dropped to 0° for all the soils at 800 °C, which contradicts our fourth hypothesis. CA > 0° measured in soils for the heating temperature of 400–800 °C, as a consequence of the small SOC contents due to the incomplete combustion of SOC, is a novelty of this study as we did not find any paper presenting CA vs. heating temperature relationships. Statistically significant differences in CA (median of CA values for temperatures 20–500 °C = the same temperature interval as for WDPT values) for soils from 1- and 10-year abandoned fields and for soils from 1- and 30-year abandoned fields were observed. A statistically significant difference in pH between 10- and 30-year abandoned fields was not found (Figure 6).

4. Discussion

It was found that all the soil properties studied were significantly affected by heating, and SOC decreased in all our soils for the heating temperature from 20 to 600 °C, but the small (0.1–0.7%) SOC contents were registered in soils heated to 600–900 °C. Badía and Martí [32] found SOC losses of approximately 90% in different Ah horizons heated 30 min to 500 °C, while heating to 250 °C caused a reduction of approximately 15–20%. The first value is slightly higher than the SOC losses we recorded when heating soil samples to 500 °C, but the second value agrees with our findings.

Our results are almost consistent with those of Martínez et al. [12], who found that soil organic matter (SOM) content decreased as the heating temperature increased. From 700 $^{\circ}$ C onward, practically no organic matter remained in their soils. However, in their soils with lower initial levels (\sim 1.9%), SOM was reduced to almost zero at 500 $^{\circ}$ C. As SOC is about 0.6 SOM, SOC in soil samples from the experimental site S1 should have decreased to almost zero at 500 $^{\circ}$ C but did not (SOC was about 0.4%).

It should be noted that complete combustion can occur only with unlimited access to oxygen. During the topsoil burning, access to oxygen is limited, which makes the combustion incomplete—on average, 50% of the SOC accumulated in the soil is burned during a fire [33]. Incomplete combustion explains that the residual 10–20% of the initial SOC remained in the soil samples (most likely in the form of pyrolytic carbon) even after heating at a temperature of $900\,^{\circ}\text{C}$.

An increase in pH(H_2O) was observed in all our soils for the heating temperature higher than 300 °C. When the soil is heated to temperatures around 300 °C, decarboxylation of humic and fulvic acids occurs, which reduces the acidic character of soil organic matter [34]. In Podzols with 85–86% of sand, Úbeda et al. [35] observed a large increase in pH at 300 °C, reaching the highest pH values at a heating temperature higher than 450 °C. However, Martínez et al. [12] found that pH increased with the heating temperature to 300–500 °C and then decreased to 900 °C, which is not consistent with our results. Negri et al. [36] observed an increase in pH with the heating temperature in an interval from 200 °C to 300 °C associated with a reduction in WDPT.

In this study, an increase in WDPT with the heating temperature was observed in an interval from 50 °C to 300 °C. Novák et al. [9] observed an increase in WDPT with increasing heating temperatures in an interval from 50 °C to 250 °C and a dramatic drop to 0 for the heating temperature of 300 °C in sandy soil under grassland in southwest Slovakia. Negri et al. [36] observed an increase in WDPT with increasing heating temperatures in

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an interval from 50 °C to 200 °C and a dramatic drop to 0 for the heating temperature of 250 °C in Regosols and Cambisols. Perera et al. [37] found that both the persistence and severity (estimated using the Molarity of an Ethanol Droplet test) of SWR of aggregates from sandy soil decreased with the heating temperature and all aggregates were wettable once exposed to 250 °C.

An increase in CA with the heating temperature was observed in all our soils in an interval from 50 °C to 300 °C. The minimum CA values in 10- and 30-year abandoned fields did not drop to 0 even at 750 °C, which means that the severity of SWR does not disappear similar to the SWR persistence at temperatures around 300–400 °C. In our opinion, this residual SWR is a consequence of the remaining SOC due to incomplete combustion. We did not find any paper presenting CA vs. heating temperature relationships, and therefore, generalization of our results is impossible. Negri et al. (2021) mentioned that CA measured in Regosols and Cambisols under European beeches and Scots pines did not exceed 120° and presented a correlation between WDPT and CA in the two datasets. Samburova et al. [38] found that all silica sand samples treated with smoke (fire emissions) or fulvic acids showed WDPT > 81 s, and contact angle (CA) between 78° and 87°, while the untreated sand samples showed WDPT < 0.5 s, and CA = 48°.

5. Conclusions

The present study evidenced that all the soil properties studied were strongly affected by heating. A drop in SOC was observed in all the soils for the heating temperature from 20 to 600 °C. Due to the incomplete combustion of SOC, a small (0.1–0.7%) SOC content was recorded even in soils heated to 600–900 °C. An increase in pH(H₂O) was observed in all the soils for the heating temperature higher than 300 °C. Soil from the experimental site S1 was wettable (WDPT < 5 s) for all of the heating temperatures. WDPT vs. heating temperature relationships for the soils from the experimental sites S2 and S3 were more complex. After a decrease caused by a heating temperature of 50 °C, an increase in WDPT for the heating temperature from 50 °C to 300 °C (for S3 soil) and 350 °C (for S2 soil) was registered. Finally, the WDPT dramatically dropped to 0 for the heating temperature of 350 °C (for S3 soil) and 400 °C (for S2 soil). CA started to decrease at 300 °C in all the soils and dropped to 0° for all the soils at 800 °C. CA > 0° measured in soils for the heating temperature of 400–800 °C, as a consequence of the small SOC contents due to the incomplete combustion of SOC, is a novelty of this study which demonstrates that CA is more sensitive to the changes in subcritical water repellency than WDPT.

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