fire

Article

The Effects of Burning Intensity on the Soil C-Related Properties and Mineralogy of Two Contrasting Forest Soils from Chilean National Parks

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Abstract

Forest fires alter multiple soil properties, from those related to the carbon cycle to mineralogy; however, the responses of various soils to thermal impact remain unclear. This study examined the impact of fire-induced heating (300, 600, and 900 °C) on the properties of two contrasted soils (Andisol and Inceptisol) with regard to soil organic carbon (SOC), total organic carbon (TOC), dissolved organic carbon (DOC), recalcitrant organic carbon (ROC), soil pH, electrical conductivity (EC), soil water repellency (SWR), soil aggregate stability (SAS), and mineralogy using X-ray diffraction (XRD). SOC and TOC decreased as temperatures increased, with a more pronounced decrease in Andisol (90% loss) than in Inceptisol (80% loss). DOC and SWR peaked at 300 °C but disappeared above 600 °C. Further, ROC increased at 300 °C in both soils, but behaved differently at higher temperatures, remaining stable in Inceptisol and being eliminated in Andisol. Soil pH increased at 600 and 900 °C; meanwhile, EC increased progressively in Andisol but peaked at 300 °C in Inceptisol. SAS remained high in both soils (between 85 and 95%) despite heating. The mineralogical analysis demonstrated how heating induced transformations in iron minerals into more oxidized forms (as hematite and maghemite) in the Andisol, while clay minerals and gibbsite decreased feldspar and quartz accumulation promotion in the Inceptisol. In summary, the initial properties of each soil influenced their respective responses to fire.

Keywords: heating; Inceptisol; Andisol; organic carbon fractions; SWR; mineral transformations

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1. Introduction

Fire is a frequent phenomenon impacting ecosystems globally, with 300 to 400 million hectares of land affected each year [1,2]. It represents one of the most widespread natural disturbances, playing a key role in many forest ecosystems, including the temperate rainforests of southern Chile, where it regulates carbon dynamics [3]. The soils in these landscapes are mainly of volcanic origin, and they exhibit high phosphorus retention and contain substantial organic matter complexed with metals, which enhances their ability to sequester carbon [4,5]. Forest fire regimes are being modified by human activities and climate change, and the frequency and severity of wildfires are expected to increase, raising concerns about the survival of existing mature forest ecosystems [4,5]. Fires alter soil properties, particularly in the topsoil horizon, where organic matter and microbial activity are concentrated. These effects depend on the intensity and duration of the fire, the environmental conditions in which it occurs, and the initial characteristics of the soil [6–9].

The first-order or direct effects of fire on the soil are brought about by an input of extreme heat into the surface horizons of the soil [10–12], commonly leading to the loss and transformation of soil organic matter (SOM), shifts in hydrophobicity, and structural changes in soil aggregation, typically in the form of aggregate destabilization. However, in specific cases, high temperatures may promote reaggregation through the recrystallization of Fe and Al oxides, particularly in soils rich in these components [13–15]. In general, total organic matter content tends to decline progressively with increased heating. In addition, surface clay can also undergo dehydration and collapse during fire events, losing key physicochemical properties [16–18]. Mineral transformations may occur at high temperatures (above 350 °C), although some changes are triggered at lower thresholds depending on heating duration and mineral composition [16,19,20]. The partial combustion of organic material promotes the formation of hydrophobic layers that reduce water infiltration and enhance runoff [21,22]. These physicochemical changes can ultimately compromise soil structural integrity, increase erosion susceptibility and hinder post-fire vegetation recovery, especially in sloped or fragile ecosystems [23–25].

One method of detecting the effects of fire on the aforementioned soil properties is to use soil heating in the laboratory, applying a wide range of both maximum temperatures (100–900 °C), application times (from minutes to two hours), or both to sieved soil samples and to undisturbed soil monoliths [23–30]. This study aims to evaluate how heating (at 300, 600, and 900 °C, for 20 min) two contrasting soils from Chilean temperate forests affects their properties. For this purpose, the temperatures have been chosen (300, 600 and 900 °C applied for 20 min) to mirror those found at the soil/litter interface of the most severe wildfires and pile burns [26,27]. This approach allows us to isolate thermal effects under controlled conditions across contrasted soil types and forest ecosystems. We hypothesize that Andisol, which contains abundant amorphous mineral phases, will experience greater carbon losses and intensified formation of iron–aluminum oxides at high temperatures. In contrast, Inceptisol, dominated by crystalline minerals, is expected to undergo less severe organic decomposition and more gradual mineral reorganization. These divergent responses are anticipated to yield distinct outcomes for water repellency, structural stability, and soil reaction, ultimately shaping the soil's carbon stabilization potential, which plays a critical role in maintaining ecosystem functions and supporting post-fire resilience. Understanding these differences is essential for developing soil-specific fire management strategies.

2. Materials and Methods

2.1. Sampling Sites

We selected two soil types from national parks in central Chile that were developed on different parent materials and in different temperate rainforests (Table 1).

Table 1. Some characteristics of the experimental soils used (Ah horizon, 0–20 cm).

	Experimental Soil	
Order	**Inceptisol**	**Andisol**
Soil subgroup ([5])	Andic Dystrudept	Typic Hapludand
Location	Nahuelbuta National Park	Puyehue National Park
Parent material	Granodiorite	Basaltic-Andesitic-scoria
Main clay type	Kaolinite	Allophane
Textural class (USDA)	Loam	Sandy clay loam
pH (1:2.5)	4.8 ± 0.1	4.8 ± 0.2
Total Organic Matter (%)	27.3 ± 0.4	21.1 ± 1.1
Plant canopy	*Araucaria araucana*, *Nothofagus pumilio*	*Nothofagus dombeyi*

We sampled an Andic Dystrudept, belonging to the order Inceptisol (hereafter referred to as Inceptisol) [28,29]. This was in Nahuelbuta National Park, under a forest canopy of *Araucaria araucana* and *Nothofagus pumilio*, on granodiorites. We sampled a Typic Hapludand, of the order Andisol (hereafter referred to as Andisol), in Puyehue National Park, under a forest canopy of *Nothofagus dombeyi* [2], both in central Chile (Figure 1).

Figure 1. Situation of Chilean national parks where soil sampling took place.

In general, the soils of the Andes are classified into four main groups: Andisols, Inceptisols, Ultisols, and Oxisols [30]. Inceptisols are one of the largest soil orders, covering nearly 15% of the global ice-free land area and supporting approximately 20% of the world's population [31]. Although Andisols represent a smaller fraction of global soil coverage (<1%), they are highly productive and support unique ecosystems across various parts of the world [5]. In Chile, both soil types are representative of the southern regions [32]. Soils derived from volcanic materials are especially important, as they account for 50–60% of the country's arable land, sustaining much of the cereal, livestock, and forestry production [33,34].

In each experimental site, a composite sample of four subsamples from the top of the Ah-horizon (0–20 cm) were taken after removing litter and duff (O-horizon). The samples were air-dried and sieved through a 2 mm mesh for soil heating treatment.

2.2. Heating Experiment

To simulate the effects of severe burning on the soils, soil subsamples (2.5 cm thick layer) were placed in porcelain containers and heated in a muffle furnace at 300 °C, 600 °C, and 900 °C for 20 min with a ramp rate of approximately 10 °C min^{-1}. Triplicate soil samples were used for each heating treatment (300, 600, and 900 °C), as well as for the unheated control. The selection of these specific temperatures and the duration of heating were based on previous studies that examined the surface soil temperatures reached during severe forest fires [12,35–38]. This approach was taken in consideration of findings suggesting that a longer exposure to moderate or high temperatures does not replicate the impact of a very high peak temperature sustained for a shorter duration on soil properties, as discussed by Thomaz and Fachin [39].

2.3. Chemical and Physical Soil Analyses

Soil organic carbon (SOC), expressed as a percentage of dry soil, was measured by wet oxidation [40], and total organic carbon (TOC) was obtained by dry combustion [41]. Dissolved organic carbon (DOC), expressed as a percentage of dry soil, was extracted with a soil–water suspension (1:5 w/v) at 80 °C, measured also via the wet oxidation method [42]. Recalcitrant organic carbon (ROC) was determined using the acid hydrolysis (HCl 6N) procedure [43]. Moreover, the soil reaction (pH) and electric conductivity (EC) were measured in 1:2.5 and 1:5 (w/v) soil–water suspensions, respectively.

The persistence of soil water repellency (SWR) was assessed through the water drop penetration time test (WDPT), which consists of applying droplets of distilled water on the soil surface and measuring the time until its complete infiltration. The analysis was conducted under laboratory conditions at controlled temperatures (20–25 °C). Drops of distilled water (~0.05 mL/drop) were applied to the 2 mm sieved soil samples, and the complete penetration time into the soil was measured. Given the wide array of values obtained via the WDPT, the SWR was categorized into five classes, defined by Bisdom et al. [44] as follows: wettable (<5 s), slightly water-repellent (5–60 s), strongly water-repellent (60–600 s), severely water-repellent (600–3600 s), and extremely water-repellent (>3600 s).

Soil aggregate stability (SAS) was determined using the wet sieving method detailed by Kemper and Koch [45] and revised by Schinner et al. [46]. This treatment emulates the forces exerted on the soil by runoff or immersion. Each soil sample (4 g) was placed in triplicate on 38 mm diameter sieves with a 0.25 mm mesh size, and then submerged and subjected to sieving action for 5 min. Subsequently, the remaining aggregates were carefully removed from the sieves, oven-dried at 105 °C, and weighed to obtain the weight of the stable aggregates and large-sized sand (>0.25 mm). Each sample was then submerged in 50 mL of 0.1 M sodium pyrophosphate decahydrate ($Na_4P_2O_7 \cdot 10H_2O$) for 2 h to disperse the stable aggregates. Finally, the samples were washed with distilled water, oven-dried at 105 °C, and weighed to obtain the weight of the sand. SAS could not be measured in the <0.25 mm sieve fraction because we used a sieve with 0.25 mm apertures. The SAS values obtained for the 1–2 mm aggregates were considered representative of the whole soil, as suggested by the method.

2.4. Mineralogical Analyses

Mineralogic changes were determined by X-ray diffraction (XRD) using a Philips PW 1710 diffractometer Eindhoven, The Netherlands) and scanned using Cu-Ka radiation, with an automatic divergence slit and diffracted-beam graphite monochromator.

The soils were then finely ground using an agate mortar and sieved to obtain a particle size < 53 µm [47]. The XRD data were stored as computer files with XPowder software [48]. For their interpretation, Brindley and Brown [49] and Joint Committee on Powder Diffraction Standards [50] were used.

2.5. Statistical Analysis

A two-factorial ANOVA was run to assess significant variance in soil response and the interactions among the two factors (temperature × soil), using experimental replicates (i.e., triplicates of the composite sample per treatment). It is important to note that true biological replication was not available, as each treatment originated from a single composite sample per soil type. As such, statistical tests were applied in an exploratory framework, and the results should be interpreted with caution. A pairwise comparison ($p < 0.05$) was also used to evaluate the statistical significance of the differences in the response variables. When assumptions of normality and homoscedasticity were met, ANOVA was applied, followed by post hoc comparisons using Tukey's test ($p < 0.05$). To satisfy the assumptions of the statistical tests, the data were subjected to normality and homoscedasticity tests and were transformed whenever necessary. In cases where even after transformation, the prerequisites were not met, the Kruskal–Wallis test was used. In addition, a principal component analysis (PCA) was performed to identify the main associations within the measured data. Prior to analysis, the data were standardized to account for differences in scale among variables. All statistical analyses were conducted using InfoStat version 2020.

3. Results and Discussion

3.1. Organic Carbon Fractions

The experimental burning of the Andisol and the Inceptisol at different temperatures (300, 600, and 900 °C) induced significant changes in soil organic carbon fractions, highlighting the impact of fire intensity on soil carbon dynamics. The response of TOC, SOC, DOC, and ROC fractions varied depending on the applied temperature and the soil type, revealing their distinct thermal decomposition patterns (Figure 2).

In both soils, TOC and SOC exhibited a marked decline with increasing temperature ($p < 0.0001$ from ANOVA), consistent with Santín and Doerr [51], who reported that SOC degradation begins around 300 °C. However, at 300 °C, SOC significantly decreased in Andisol, with losses of 38%, whereas Inceptisol did not show significant changes compared to the control. This suggests that Andisol, despite its high organic matter retention capacity, is more susceptible to thermal degradation at moderate fire intensities, leading to its decomposition at higher temperatures. In contrast, the lack of significant SOC changes in Inceptisol at 300 °C suggests a higher resistance to moderate fire intensity, possibly due to its different stabilization mechanisms. However, at 600 and 900 °C, both soils experienced a sharp decline in SOC, indicating extensive oxidation of organic compounds, aligning with previous studies on high-intensity fires [10,52,53]. Other authors found reductions in total organic matter and its fractions due to controlled burning, which in turn correlated with decreases in the microbial activity and biological functioning of the soil [54,55], or with soil erodibility increases [53].

Regarding DOC, both soils showed a significant increase at 300 °C compared to the control; 86% for the Andisol and 128% for the Inceptisol, indicating that moderate fire intensity enhanced the solubilization of organic compounds, increasing the lability of the initial organic carbon. This response is likely due to the thermal breakdown of organic macromolecules into smaller, water-soluble compounds, a mechanism previously reported by Dou et al. [56] and Ludwig et al. [57], who observed a post-fire increase in DOC with burn severity. However, at 600 and 900 °C, DOC drastically decreased in both soils, suggesting the progressive oxidation and mineralization of labile organic compounds [58]. The significant interaction between soil type and temperature indicates that DOC dynamics are influenced by both factors, with the Inceptisol showing a stronger DOC increase at 300 °C compared to the Andisol. Also, Bravo-Escobar et al. [59] found that DOC increased in a burned (4 months ago) soil (0–5 cm) of a native jarrah forest (*Eucalyptus marginata*), but decreased in an exotic pine plantation (*Pinus radiata*) in SW Australia, suggesting that the interaction between forest type and fire varies the behavior of DOC.

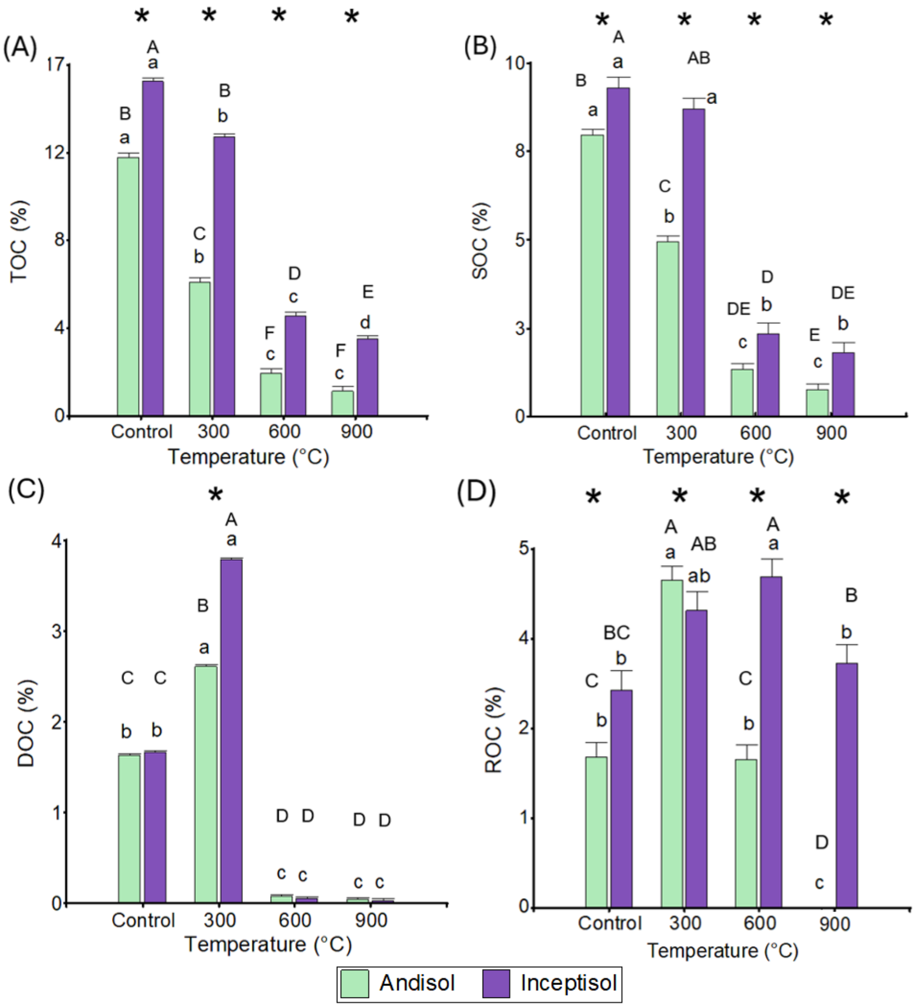


Figure 2. The effects of burning on soil organic matter (SOM) fractions. (**A**) The response of total organic carbon (TOC), (**B**) soil organic carbon (SOC), (**C**) dissolved organic carbon (DOC), (**D**) and recalcitrant organic carbon (ROC). Lowercase letters on top of the bars indicate significant differences between treatments, while asterisks indicate significant differences between soils ($p < 0.05$). Uppercase letters on top of the bars indicate significant differences within all samples when the interaction between treatment and soil was significant. In each bar, the mean ($n = 3$) and the standard deviation are represented.

Other studies found high DOC levels in soils heated to 250 °C or below when compared to unheated controls, but drastic decreases at 500 °C [60,61]. In short, these findings highlight the dynamic response of DOC to fire intensity, with a transient increase at moderate temperatures followed by depletion under extreme burning conditions, emphasizing the importance of temperature thresholds in post-fire carbon cycling. According to Gui et al. [62], SOC and DOC significantly decreased in temperate forests after fire, and this limitation of carbon supplements could be a major driver for the observed responses of soil respiration (SR) and heterotrophic respiration (HR) to fire.

ROC exhibited a distinct trend compared to other fractions in the Andisol from Kruskal–Wallis ($p = 0.0227$) and in the Inceptisol from ANOVA ($p = 0.0106$). At 300 °C, ROC increased significantly in Andisol (by about 120%), indicating the partial transformation of SOM into more recalcitrant forms. This is due to moderate fire intensity promoting the formation of thermally stabilized organic compounds. However, at 600 °C, ROC continued to increase in Inceptisol, suggesting that this soil type retained a greater proportion of fire-resistant organic compounds at this temperature. At 900 °C, ROC significantly decreased in both soils, indicating the extensive combustion of recalcitrant carbon. These results align with studies reporting that fire can enhance the formation of pyrogenic organic matter at intermediate temperatures [58], but excessive heat exposure at 900 °C leads to its destruction. The significant interaction effect between soil and temperature further highlights that the persistence of ROC varies between soils, with the Inceptisol being more resistant to high-temperature transformations. The divergent behavior of ROC between the two soil types suggests that their recovery after fire may vary between them [63].

3.2. pH and Electrical Conductivity

The results indicate that soil pH did not show significant changes at lower burning temperatures (300 °C) (Figure 3A) in the Andisol, via Kruskal–Wallis ($p = 0.0216$), or in the Inceptisol, via ANOVA ($p < 0.0001$). These findings align with previous studies' suggestions that when ashes are not incorporated into the mineral soil, pH changes remain limited [11,59,64]. However, at 600 °C and 900 °C, a significant increase in pH was observed, especially in the Andisol, indicating that high temperatures induce chemical transformations that create a more alkaline environment. On the other hand, electrical conductivity (EC) showed significant variations from ANOVA ($p < 0.0001$) (Figure 3B). In the Inceptisol, EC drastically increased at 300 °C, suggesting a rapid release of soluble salts due to organic matter combustion and clay decomposition [11,16]. However, at 600 °C and 900 °C, EC decreased, likely due to the volatilization of salts. Conversely, in the Andisol, EC progressively increased with temperature, reaching its highest value at 900 °C, reflecting a more gradual release of ions in the soil solution. This behavior has been observed in other studies, where the greater availability of soluble ions after fire is associated with organic matter combustion and oxide formation [64]. These results suggest that the mineralogical composition of the soil influences its response to fire, with the Andisol exhibiting greater chemical stability than the Inceptisol.

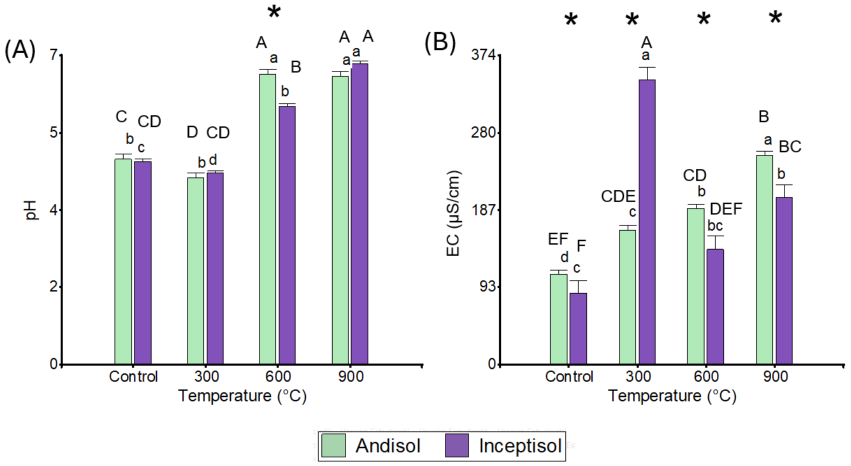


Figure 3. Burning's effects on (**A**) soil pH and (**B**) soil electric conductivity (EC). Lowercase letters on top of the bars indicate significant differences between treatments, while asterisks indicate significant differences between soils ($p < 0.05$). Uppercase letters on top of the bars indicate significant differences within all samples when the interaction between treatment and soil was significant. In each bar, the mean ($n = 3$) and the standard deviation are represented.

3.3. Soil Aggregate Stability and Soil Water Repellency

In unburned samples, the SAS was very high in both soils (>85%), and a slight but significant increase at 300 °C was observed only in the Inceptisol by ANOVA ($p < 0.0001$). At higher temperatures, the SAS remained high in both soils (Figure 4A). These findings are consistent with previous studies suggesting that soil aggregation can increase after fires when soil temperatures stay below 220 °C [65]. Although there is a reduction in SOC content, which may be attributed to mineralogical changes, Jiménez-Pinilla et al. [38] observed increased aggregation after heating at 300 °C, associated with the compaction of structural units. Similarly, Giovannini et al. [66] reported that soil gel dehydration at temperatures above 170 °C promotes aggregation. The reduction in organic matter is often accompanied by mineral transformations, such as the formation of iron and aluminum oxides or the collapse of clay minerals, which can modify surface reactivity. Despite the loss of SOC, transient increases in aggregation may occur due to the formation of inorganic cementing agents, such as iron and aluminum oxides, or due to structural compaction and gel dehydration [16]. However, Giovannini and Lucchesi [65] also noted that aggregation may remain stable after SOM combustion at 150 °C due to the transformation of cementing iron oxides. In contrast, Badía and Martí [67] observed a decrease in SAS in two semiarid soils burned at 150, 250, and 500 °C, mainly related to SOM loss.

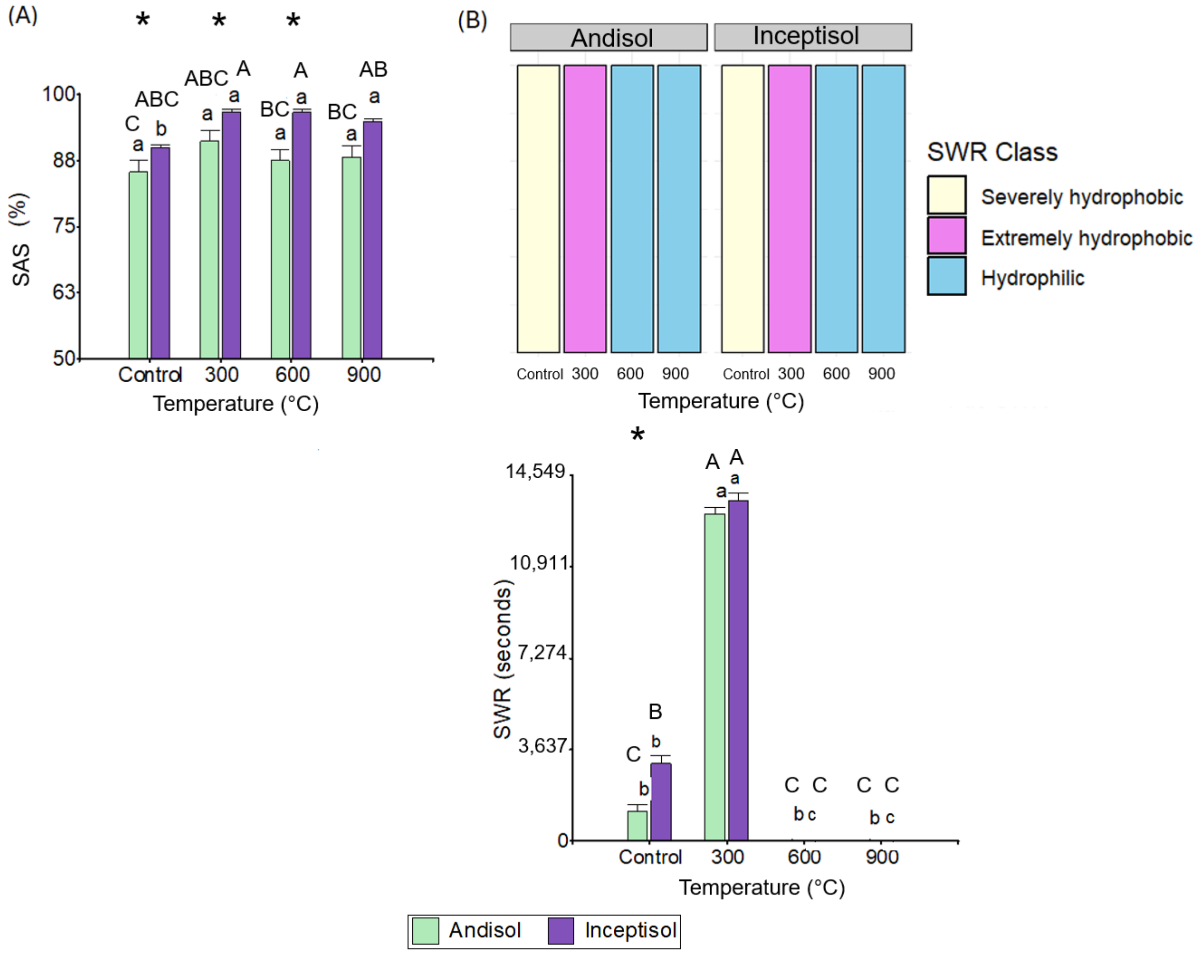


Figure 4. (**A**) Soil aggregate stability (SAS) and (**B**) soil water repellency (SWR) according to the water drop penetration time (WDPT) test for the unburned (control) and burned (300, 600, 900 °C) samples. Lowercase letters on top of the bars indicate significant differences between treatments, while asterisks indicate significant differences between soils ($p < 0.05$). Uppercase letters on top of the bars indicate significant differences within all samples when the interaction between treatment and soil was significant. In each bar, the mean ($n = 3$) and the standard deviation are represented.

SWR was significantly influenced by fire temperature from Kruskal–Wallis ($p = 0.0138$), with a clear transition observed between hydrophobic and hydrophilic states (Figure 4B). In both Andisol and Inceptisol, unburned (Control) soils exhibited severely hydrophobic properties, confirming that natural SWR is common in many soils [11,68,69]. This behavior may be related to the dominant native vegetation in these areas [68]. These hydrophobic substances can be transferred to the soil through litterfall, root exudates, or decomposition products, contributing to the development of natural water repellency even in the absence of fire disturbance. However, at 300 °C, SWR intensified to an extremely hydrophobic state, indicating the formation of hydrophobic compounds from organic matter combustion [70]. Fire can induce soil water repellency by promoting the thermal volatilization and subsequent downward migration of hydrophobic organic compounds from the upper layers of litter and SOM into deeper mineral horizons [70]. As the soil cools, these compounds condense and coat mineral particles, forming hydrophobic layers that reduce water infiltration and increase runoff potential. This behavior aligns with previous studies where low- to moderate-intensity fires promoted SWR due to the partial combustion of SOM and the accumulation of hydrophobic substances [71]. At higher temperatures (600 and 900 °C), both soils became hydrophilic, indicating the destruction of hydrophobic compounds through complete SOM combustion [52]. This pattern suggests that fire intensity plays a key role in determining post-fire SWR, with intermediate temperatures enhancing repellency and extreme temperatures eliminating it. The findings are consistent with studies reporting that high-severity fires lead to the loss of SWR due to organic matter volatilization [70]. The differences between Andisol and Inceptisol suggest that vegetation type, mineralogical composition, and organic matter content modulate SWR dynamics, reinforcing the link between SOC content and post-fire hydrophobicity.

Overall, fire intensity, rather than soil type, was the dominant factor influencing hydrophobicity and soil aggregation, with moderate burns tending to enhance both properties, while extreme burns led to the complete loss of water repellency [70]. These post-fire changes in physical properties are critical because increased soil water repellency and altered aggregation can reduce infiltration, promote surface runoff, and significantly elevate soil erosion risks, especially on sloped terrains [21,22,72]. Moreover, these alterations may hinder vegetation regrowth by limiting water availability and destabilizing the seedbed, thus slowing ecosystem recovery [73].

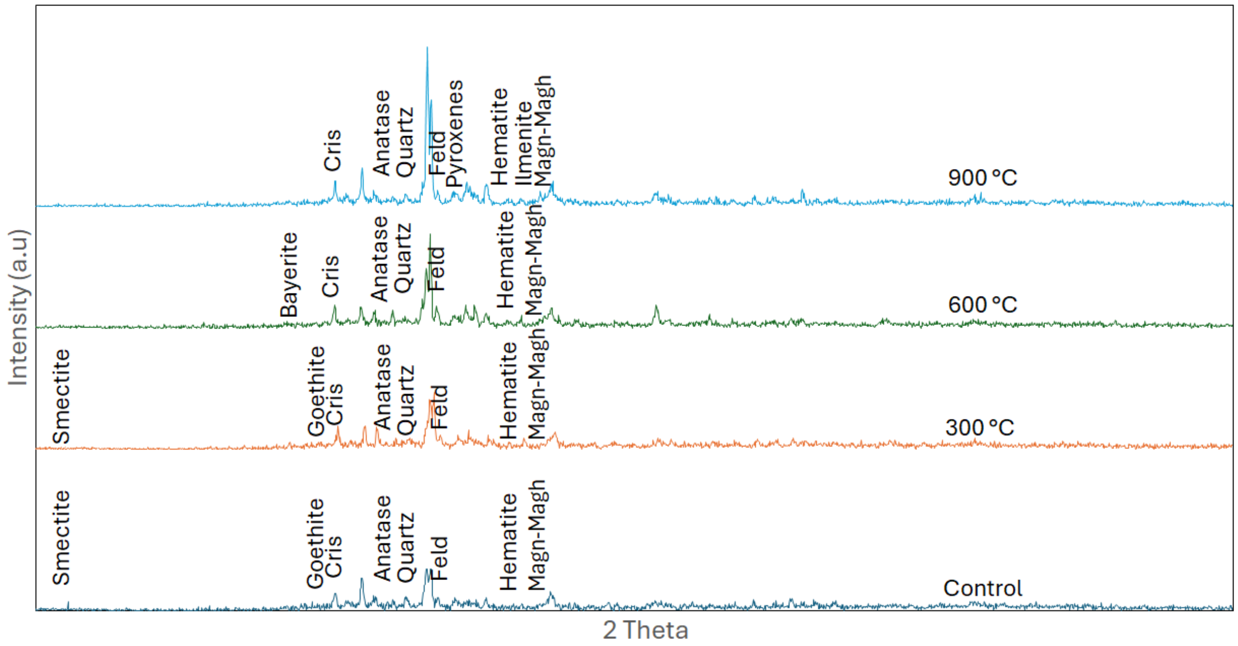
The mean values for all measured soil properties across fire intensity treatments (control, 300, 600, and 900 °C) for both Andisol and Inceptisol were compiled and are shown in Supplementary Table S1.

3.4. Mineralogical Modifications

The heating of Andisol and Inceptisol revealed transformations in their mineralogy, with notable differences in the stability of certain minerals (Figure 5).

In Andisol, smectite, a clay mineral with high cation exchange capacity, completely disappeared at 600 °C, after a very light initial reduction at 300 °C, indicating a rapid thermal transformation [74]. Similarly, goethite decreased about 7% at 300 °C and then vanished at 600 °C, consistent with previous studies indicating its conversion to hematite in this temperature range [75]. The increase in feldspar content from 31% to 54% at 600 °C, and up to 63% at 900 °C, suggests the partial melting and crystallization of stable aluminosilicate phases [76]. The formation of hematite and magnetite–maghemite at 900 °C confirms the oxidation of iron minerals, driven by the decomposition of goethite at high temperatures [77]. In contrast, the Inceptisol showed a transformation in clay minerals and a greater accumulation of quartz, indicating more severe structural degradation. Gibbsite, initially present at 2.5% in the control, completely disappeared at 600 °C, consistent with its dehydration in this temperature range [78]. The Inceptisol developed γ-alumina, reflecting aluminum mineral transformations linked to gibbsite breakdown [37]. The loss of muscovite and chlorite marked the instability of secondary minerals, promoting the accumulation of feldspar and quartz. By 900 °C, quartz content increased from 64% to 76%, suggesting the recrystallization of amorphous silica and the loss of less stable phases. Additionally, the presence of anatase at high temperatures indicates that titanium oxides are highly resistant to combustion and may contribute to soil thermal stabilization [79].

(A) Andisol



(B) Inceptisol

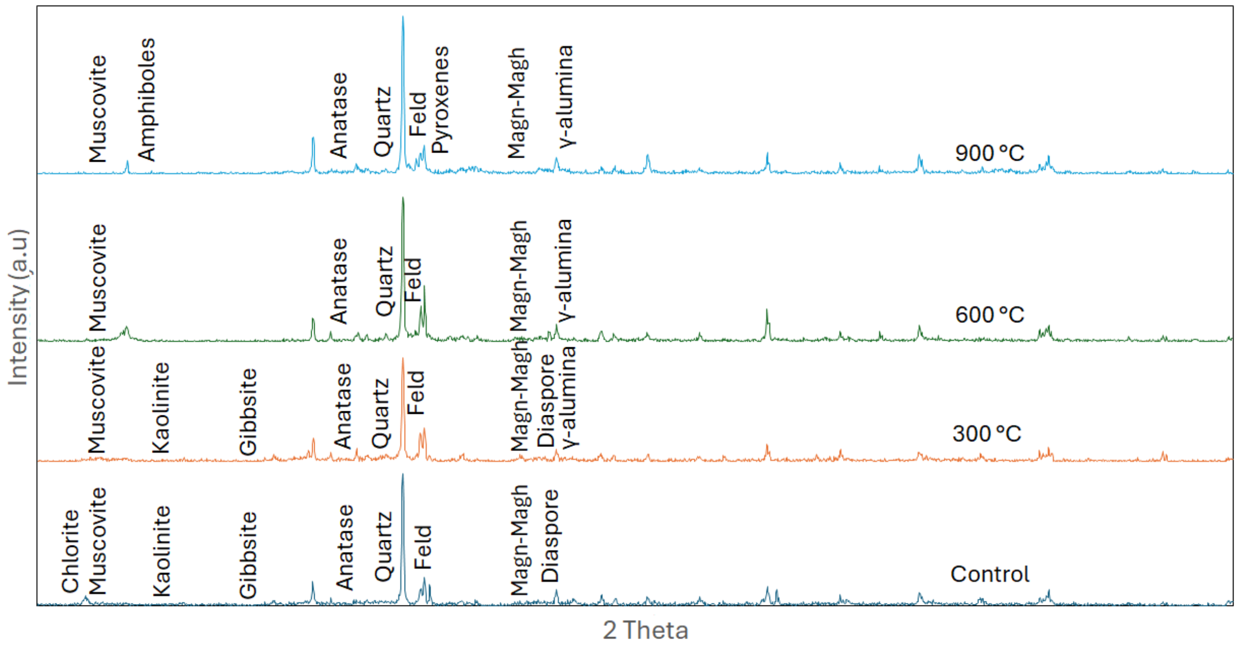


Figure 5. X-ray diffraction (XRD) patterns of the control (unheated) and heating temperature treatments (300, 600, and 900 °C) of two soils: (**A**) Andisol and (**B**) Inceptisol. Abbreviations: Cris: cristobalite; Feld: feldspar; Mgn-Magh: magnetite–maghemite.

These results suggest that the thermal resistance of the Inceptisol is primarily due to its high quartz and feldspar content, whereas the Andisol undergoes fewer structural changes in crystalline minerals but could suffer greater loss of amorphous minerals, as described in previous studies on volcanic soils [80]. Despite the thermal transformation of goethite and smectite in the Andisol, the stability of hematite and feldspar indicates that this soil retains part of its mineral structure at high temperatures. In contrast, Inceptisol, losing its key clay minerals while increasing in quartz, appears more structurally fragile under heating, potentially reducing its carbon stabilization capacity. The loss of secondary and non-crystalline minerals, such as goethite and gibbsite, will reduce both soil surface reactivity and the protective microsites and absorbent surfaces that promote microbial colonization and the stabilization of organic compounds [81,82]. Conversely, the relative enrichment in thermally stable phases such as quartz, feldspar, hematite, and γ-alumina reflects a mineralogical assemblage that is less chemically active and less conducive to microbial growth [16,37].

3.5. Spearman Correlations Among Soil Properties

The correlation coefficients between various physicochemical properties in the two studied soils were assessed by Spearman correlation (Figure 6), along with their significance values (p-values).

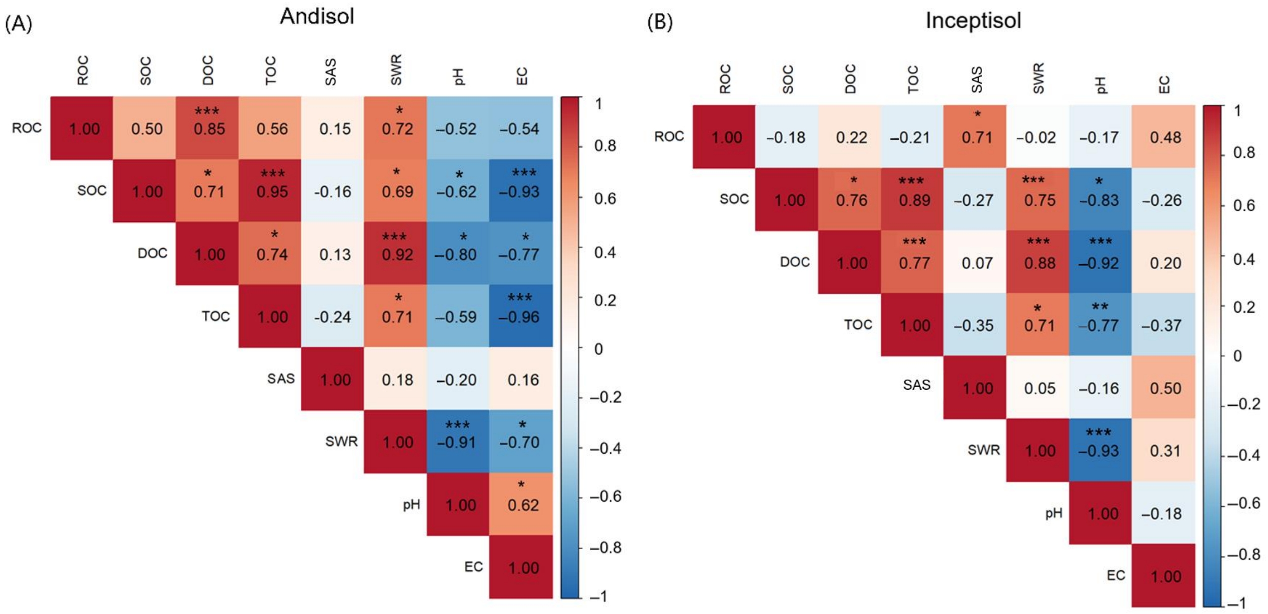


Figure 6. Spearman correlation matrices for two soil orders: (**A**) Andisol and (**B**) Inceptisol. The scale ranges from −1 (blue) to +1 (red). The variables include recalcitrant organic carbon (ROC), soil organic carbon (SOC), dissolved organic carbon (DOC), total organic carbon (TOC), aggregate stability (AS), soil water repellency (SWR), pH, and electrical conductivity (EC). Asterisks denote correlation values: * $p < 0.05$, ** $p < 0.01$, and *** $p < 0.001$.

In the Inceptisol, a strong positive correlation exists between TOC and DOC (r = 0.77, $p < 0.001$). This relationship suggests that soils with elevated organic carbon content have a greater potential to release dissolved carbon, likely due to enhanced decomposition processes and the mobilization of organic compounds. SWR and DOC showed an extremely strong correlation (r = 0.88, $p < 0.001$), suggesting that increased DOC could be related to higher water repellency. The labile fraction of SOM includes soluble compounds such as lipids, waxes, fatty acids, and other aliphatic materials. These compounds, which are part of the non-humified or only partially transformed organic matter, are more readily mobilized and can contribute to water repellency in soils [83]. pH has significant negative correlations with DOC (−0.92), TOC (−0.77), and SWR (−0.93), indicating that as pH increases, these parameters decrease. SOC and SWR are positively correlated (r = 0.75, $p < 0.001$), suggesting that the accumulation of organic carbon contributes to the formation of hydrophobic coatings on soil particles, reducing the soil's ability to retain water [84].

In the Andisol, DOC and ROC had a strong positive correlation (r = 0.85, $p < 0.001$), indicating that increased dissolved carbon was also associated with an increase in the recalcitrant fraction. Andisols are known for their high capacity to adsorb and retain organic matter, mainly due to the presence of minerals such as allophane, imogolite, and iron and aluminum complexes. These organo-mineral associations promote the stabilization of organic carbon and lead to the accumulation of durable fractions [85]. However, when exposed to elevated temperatures, such as those occurring during a fire, part of this stabilized organic matter may be thermally altered or released, contributing to an increase in dissolved carbon [86,87]. pH presented significant negative correlations with SWR (−0.91) and DOC (−0.8). EC and TOC had a strong negative correlation (r = −0.96, $p < 0.001$), suggesting that higher EC corresponds to lower organic carbon content. Since organic matter often contributes to cation retention, it reflects the effect of fire-induced heating. In the Andisol, rising temperatures promote the degradation and volatilization of organic matter, resulting in a marked decrease in TOC. Simultaneously, this thermal decomposition leads to the release of previously complexed or adsorbed cations, increasing their concentration in the soil solution and thus raising EC [26]. This mineralization-induced release of soluble ions explains the inverse relationship observed between TOM and EC in burned soils.

3.6. Principal Component Analysis

In the Andisol, the primary sources of variation were changes in SOC, TOC, DOC, and SWR along one axis, and pH and EC along the other (Figure 7). The distribution of variables suggested that higher temperatures (600 °C and 900 °C) were associated with an increase in pH and EC, while lower temperatures (Control and 300 °C) were linked to SOC and TOC. Notably, 300 °C was positioned relatively close to the control, indicating that moderate fire intensity did not drastically alter soil properties, likely due to the stabilizing effect of amorphous minerals, such as allophane and imogolite, which can buffer against heat-induced changes at moderate temperatures [17]. However, ROC and SWR increased at 300 °C, suggesting early-stage organic matter transformation and increasing hydrophobicity (see Figure 4B) [13,25].

In the Inceptisol, the control and 300 °C treatments were positioned farther apart in the principal component analysis (PCA) compared to the Andisol, suggesting that the Inceptisol exhibited a stronger multivariate response to moderate heating. However, this does not necessarily imply lower thermal resistance in terms of organic carbon stability, as the Andisol showed greater absolute losses of SOC and TOC at 300 °C. According to dos Santos et al. [88], interactions between clay minerals such as gibbsite and humic fractions like fulvic acid (FA) can form organo-mineral complexes that are relatively resistant to degradation compared to free SOM. Nevertheless, their protective effect decreases at higher temperatures, as observed in this study, where gibbsite was absent at 600 °C (Figure 5B). Therefore, the apparent greater persistence of ROC in the Inceptisol at elevated temperatures may be more closely related to pyrogenic inputs from vegetation or site-specific factors rather than to mineral protection alone. Also, in the Inceptisol the PCA showed that 300 °C was more closely associated with increased SWR and DOC, suggesting an early-stage breakdown of organic matter and enhanced soil hydrophobicity (Figure 4B). The variables pH and EC shifted significantly at higher temperatures (600 °C and 900 °C), reflecting mineral transformations and increasing the solubility of ions [68]. These results indicated that the Inceptisol experienced a more pronounced alteration at moderate fire intensities, while the Andisol displayed a more gradual transition.

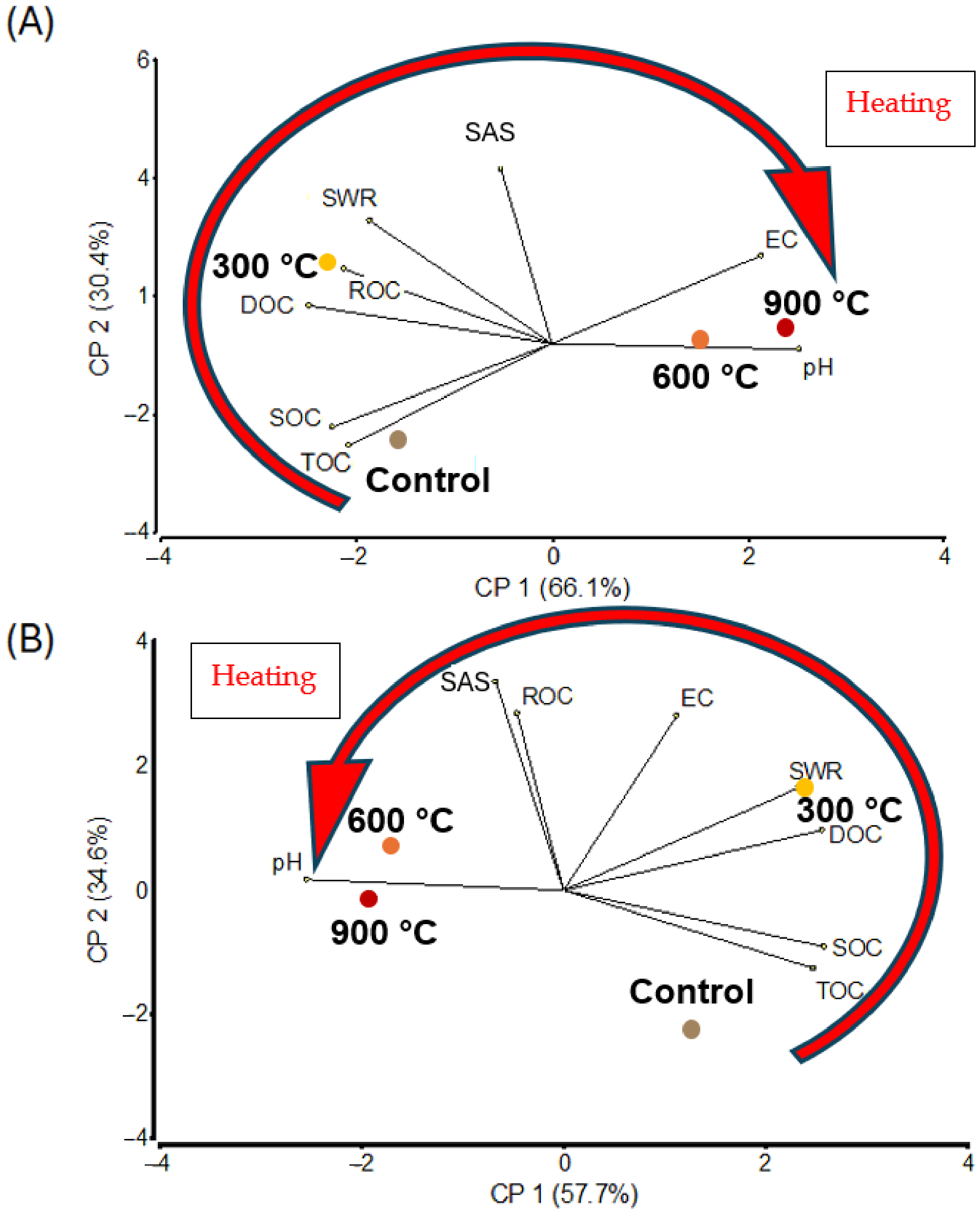


Figure 7. Principal component analysis (PCA) of Andisol (**A**) and Inceptisol (**B**). The first two components (CP1 and CP2) explain most of the total variance. Each soil sample was displayed at four heating levels: control, 300 °C, 600 °C, and 900 °C. The vectors represent aggregate stability (AS), soil water repellency (SWR), recalcitrant organic carbon (ROC), dissolved organic carbon (DOC), total organic carbon (TOC), soil organic carbon (SOC), pH, and electrical conductivity (EC). The red arrows indicate how each soil shifts in response to increasing temperatures.

Therefore, PCA biplots (Figure 7) illustrated these divergent trajectories, confirming that moderate heating caused increases in DOC and SWR, whereas higher temperatures induced elevated pH and EC and reduced organic fractions [88].

These findings underscore the importance of soil mineralogy and organic fraction type in evaluating the impact of fire on carbon storage and soil properties. The capacity for post-fire soil recovery depends on both the extent of organic matter preservation and the stability of the mineral phases. Future work could extend these observations to natural burn conditions, where heat distribution, moisture, and vegetation composition add variability. Understanding these mechanisms will inform fire management and guide soil restoration in temperate forested regions with contrasting soil orders.

4. Conclusions

The temperature exerted a dominant influence on soil organic carbon fractions and mineralogy in the two studied soils (Andisol and Inceptisol) under temperate rainforests. Both TOC and SOC decrease progressively with heating increase but do so differently in both soils. Andisol exhibited greater carbon losses as temperature increased. At 300 °C, DOC, ROC, and SWR increased, but decreased with higher temperatures; in contrast, the highest (600 and 900 °C) temperatures increased pH and ions in solution (EC). The disappearance of minerals such as smectite and goethite, together with the formation of hematite and magnetite at higher temperatures, indicated more intense mineral transformation and iron oxidation processes in the Andisol. In contrast, Inceptisol, although it showed abrupt changes at 300 °C in other evaluated properties, experienced lower total SOC and TOC losses and retained structurally stable crystalline phases such as quartz and feldspar at higher temperatures. However, it also lost clay-associated minerals such as gibbsite. The relative persistence of ROC in the Inceptisol at 600 °C suggests that carbon stabilization in this soil may be more closely related to pyrogenic organic inputs derived from vegetation, rather than progressive mineral reorganization. Overall, the findings largely support the proposed hypothesis. Nevertheless, future research should incorporate analytical techniques capable of identifying amorphous minerals to directly confirm their thermal transformation. This is essential to better understand the mechanisms of carbon protection or release under fire conditions, particularly in volcanic soils rich in non-crystalline phases.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/fire8070277/s1, Table S1: Changes in the properties of the two soils subjected to different temperatures: control or unheated, 300, 600 and 900 °C (mean values ± standard deviation).

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