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Alterations in aggregate characteristics of thermally heated water-repellent soil aggregates under laboratory conditions

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Abstract: The heat generated during wildfires modifies soil characteristics, including soil water repellency (SWR) and the water stability of aggregates, which are known to be interrelated. SWR lowers the rate of water entry into aggregates, minimizing aggregate disruption and subsequent erosion. This study aimed to examine these aggregate characteristics (SWR, water stability of aggregates) of thermally heated water-repellent soil aggregates under laboratory conditions. Water-repellent aggregates were collected from *Eucalyptus grandis* forest soil separately from four soil depths (0–5, 5–10, 10–15, and 15–20 cm) with varying initial repellency levels. Using an automated programmable muffle furnace, aggregates were separately exposed to three heating temperatures, T_H (150, 200, 250 °C), three rates of heating (speed of rising temperature to reach relevant T_H), R_H (200, 400, 800 °C h^{-1}), and three durations of exposure to relevant T_H, E_D (30, 60, 120 min). The molarity of an ethanol droplet test was used to measure the contact angle (contact angle>90°). The water drop penetration time (WDPT) was also measured. The SWR of aggregates declined with the increasing T_H and E_D. All aggregates were wettable once exposed to 250 °C. At the lowest T_H and E_D (150 °C, 30 min), the contact angle was <90° only in the least repellent aggregates collected from 10–15 and 15–20 cm depths. Although R_H indicated the least influence on the measured parameters, the slowest R_H (200 °C h^{-1}) caused a comparatively greater decline in SWR. Water stability of aggregates increased with heating irrespective of decreasing SWR. Further investigations on heat-induced changes in organic compounds at molecular levels would be necessary to understand the theories for the behavior of aggregates.

Keywords: *Eucalyptus grandis*; Laboratory heating; Water repellency; Water stability of aggregates.

INTRODUCTION

Soil water repellency (SWR) reduces the affinity of soil for water and hinders spontaneous wetting. It is a global phenomenon that occurs in different climates varying from subarctic to tropical and in a wide range of soils (Benito et al., 2019; Iovino et al., 2018; Kobayashi and Shimizu, 2007; Leelamanie and Nishiwaki, 2019; Leelamanie et al., 2021; Lin et al., 2006; Piyaruwan and Leelamanie, 2020). The presence of organic materials in the soil, as coatings on soil mineral grains and intermixed materials, causes water–repellent conditions. Many factors influence water-repellent conditions, including the content and the composition of organic carbon (Atanassova and Doerr, 2010; Dao et al., 2022), fungal activities, pH (Lin et al., 2006), temperature (Zavala et al., 2010), humidity levels (Whelan et al., 2014), and soil moisture levels (Caltabellotta et al., 2022; Kajiura et al., 2012). The immediate impact of SWR is the hindrance of water infiltration into the soil, consequently causing increased overland flow and topsoil erosion (Butzen et al., 2015).

One of the positive impacts of SWR is its contribution to the improvement of the stability of aggregates. SWR hinders the water entry rate into the aggregates, lowering the pressure buildup in intra–aggregate pore spaces and enhancing the ability of aggregates to resist disruption (Piyaruwan and Leelamanie, 2020). As a result, the stability of aggregates in water-repellent soils is reported to be relatively high (De Gryze et al., 2006; Piyaruwan and Leelamanie, 2020). Both SWR and aggregate stability influence the soil erodibility (Badía-Villas et al., 2014), which expresses the susceptibility of soil to be detached and consequently be transported away (with water, wind, or ice). A well-developed structure and resistant or stable aggregates in topsoil lowers the erodibility of soil (Martínez-Murillo et al., 2020).

SWR is strongly associated with specific tree species that consist of significant quantities of polar waxes and/or resins. Eucalyptus (*Eucalyptus globulus; Eucalyptus grandis*) (Piyaruwan and Leelamanie, 2020; Walden et al., 2015), Pine (*Pinus pinaster, Pinus halepensis*, *Pinus sylvestris, Pinus caribaea*) (Iovino et al., 2018; Lichner et al., 2013), Japanese cypress (*Chamaecyparis obtusa*), Japanese cedar (*Cryptomeria japonica*) (Kobayashi and Shimizu, 2007; Leelamanie and Nishiwaki, 2019), and Casuarina (*Casuarina equisetifolia*) (Leelamanie, 2016; Lin et al., 2006) are few examples for such tree species. Because of the waxy or oily nature of their plant debris, plant species such as conifer and Eucalyptus are highly flammable and vulnerable to wildfires (Bernier et al., 2016; Scarff and Westoby, 2006). Furthermore, flash fuels, supplied by litter materials with waxy flammable nature cause a more rapid rise in soil temperature, or rapid heating rates (Bruns et al., 2020).

Wildfires disrupt forests altering the physical, chemical, and biological characteristics of the underlying soils. The heat generated during a wildfire increases the soil temperature (Fajković et al., 2022; Li et al., 2022; Neary et al., 1999 and references therein), which has a significant influence on the SWR (Caltabellotta et al., 2022; Doerr et al., 2004; García-Corona et al., 2004; Hološ et al., 2022; Novák et al., 2009; Plaza-Álvarez et al., 2018; Zavala et al., 2010). There are pieces of evidence available to show that the heat generated by wildfire can either increase, decrease, or eliminate the SWR conditions (Doerr et al., 2006; Zavala et al., 2010). The impact of heat on underlying soils depends on many factors such as the intensity and the severity of fire (Doerr et al., 2006), associated peak soil temperature (Zavala

et al., 2010), the duration of exposure to that temperature peaks (Dao et al., 2022; Doerr et al., 2004), rising rate of temperature (Bruns et al., 2020), and moisture levels during heating (Zavala et al., 2010). However, the reports on temperature levels and conditions that are associated with increment, decline, or elimination in SWR are contradictory. Several reports show that SWR increases with increasing temperature for the ranges of 100–150 °C (Zavala et al., 2010), 50–150 °C (Dao et al., 2022; Doerr et al., 2005), and 25–220 °C (García-Corona et al., 2004). Doerr et al. (2005) reported that SWR starts to decrease at 200 °C, while Zavala et al. (2010) stated that SWR decreases at about 250 °C. Other studies report 210 °C (Bryant et al., 2005), 260 °C (Doerr et al., 2004), 300 °C (Doerr et al., 2005), 380 °C (García-Corona et al., 2004), and 400 °C (Hološ et al., 2022; Zavala et al., 2010) as the temperatures that cause the elimination of SWR. In addition to SWR, the stability of soil aggregates is another important soil property that tends to alter due to the heat generated during wildfires (Mataix–Solera et al., 2011 and references therein; Zavala et al., 2010). Soil erosion is considered a serious consequence of wildfire although its effects on the stability of aggregates are far from clear (Varela et al., 2010). A wildfire incidence can enhance or deteriorate the stability of aggregates through the removal of the vegetation and litter during the process of burning (Arcenegui et al., 2008; Mataix-Solera and Doerr, 2004; Varela et al., 2010). The effect of heat on the stability of aggregates depends on the heating dynamics (Guerrero et al., 2001 and references therein; Zavala et al., 2010) as well as on the heat-induced modifications in SWR (Arcenegui et al., 2008; García-Corona et al., 2004). According to the reported literature, aggregate stability increases when SWR increases due to heating (García-Corona et al., 2004). However, at higher temperatures, SWR levels decrease, leading to a reduction in aggregate stability (García-Corona et al., 2004; Zavala et al., 2010). Furthermore, it increases in some non–repellent soils as well due to the consequences of heating such as dehydration of soil gels between 175–220 °C and the recrystallization of Al and Fe at higher temperatures (Guerrero et al., 2001 and references therein). In addition, the stability of aggregates decreases as a result of phenomena such as the volatilization of soil organic compounds due to heating at higher temperatures above 200 °C (Zavala et al., 2010).

The information available in literature related to the direct and indirect effects of heat (temperature, duration, and rising rate) on SWR and aggregates, is contradictory or not in extensive detail. The objective of this study was to examine the effects of different heating dynamics (temperature, heating rate, and duration of exposure to heat) on SWR, and water stability of originally water-repellent soil aggregates under laboratory conditions using a *Eucalyptus* forest soil.

MATERIALS AND METHODS

Sample collection and preparation

Soil samples for the experiment were collected from a long unburned *Eucalyptus grandis* forest in Sri Lanka (06° 47′ 42.5″N 80° 58′ 00.5″E) (Figure 1). According to the Köppen-Geiger climate classification, the climate of the area falls into the Equatorial fully humid category (Kottek et al., 2006), and the soil type is Hapludults according to the USDA classification (Soil Survey Staff, 2014). The 3–5 cm thick litter layer of dried foliage and twigs was removed carefully by brushing prior to the sample collection. Sampling was conducted down to 20 cm (non-repellent/slightly water-repellent level) in four depths (0–5, 5–10, 10–15, and 15–20 cm) from three replicated points to investigate the processes that would occur through the soil profile (Lichner et al., 2007). The basic characteristics of the collected soil are presented in Table 1.

Since the collected soil was rich in granular structure (crumbs), aggregates with 3–5 mm diameter were selected for this study. Aggregates were manually sorted from the collected soil samples and were air-dried under 65±5 % RH and 28±2 °C temperature prior to further analysis. Aggregates were turned periodically to ensure homogenous drying during the air-drying process. After air drying, aggregates with approximately similar weights were selected for the study, discarding those with large stones and root fragments.

Heat treatments

The heating temperature (to represent the strength or the severity of a fire), duration of exposure (to represent the persistence of a fire), and the rates of heating (to represent the rising rate of temperature influenced by the availability of flash fuels) were considered as the three heating parameters for the experiment. Heat treatments (27) consist of three heating temperatures, T_H (150, 200, 250 °C), three rates of heating (speed of rising temperature to reach relevant T_H), R_H (200, 400, 800 °C h^{-1}), and three durations of exposure to relevant T_H, E_D (30, 60, 120 min).

Table 1. Basic properties of tested soil.

Property	0–5 cm	5–10 cm	10–15 cm	15–20 cm	Method	Reference
Degree of SWR (°)	103±1	99±1	91±1	90±0	Molarity of an ethanol droplet (MED) test	Poulenard et al., 2004
Persistency of SWR (s)	7200±0	510±17	15±3	2±1	Water drop penetration time (WDPT) test	Chenu et al., 2000
Bulk density (g cm^{-3})	0.95±0.03	1.01±0.09	1.03±0.05	1.18±0.03	Undisturbed core method	Blake and Hartge, 1986a
Particle density (g cm^{-3})	2.50±0.09	2.43±0.14	2.14±0.02	2.43±0.14	Pycnometer method	Blake and Hartge, 1986b
Porosity (%)	61.77±0.02	59.59±0.04	52.17±0.02	52.70±0.02	Numerical method	
Air–dried moisture (%)	7.33±1.85	6.83±0.61	5.36±1.20	4.01±1.57	Gravimetric method	Reynolds, 1970
Sand (%)	82.1 ± 0.1	79.4 ± 0.1	76.7 ± 0.2	75.14±1.48	Hydrometer method	Gee and Bauder, 1986
Silt (%)	5.1 ± 0	6.9 ± 1.5	10.3 ± 0.1	10.29±2.10		
Clay (%)	12.8 ± 0	13.7 ± 1.5	12.9 ± 0.1	14.57±1.21		
Texture	Sandy loam	Sandy loam	Sandy loam	Sandy loam		
Organic matter (%)	12.73±1.16	10.46±0.89	9.18±0.81	7.49±0.81	Loss on ignition method	Schumacher, 2002
pH	4.13±0.06	4.13±0.06	4.37±0.25	4.27±0.12	HachsensION 1 pH meter	
EC (mS m^{-1})	0.05±0.02	0.05±0.01	0.06±0.01	0.07±0.01	Hanna Instruments, HI 9812, a.k.a Combo 1 EC meter	

Fig. 1. Soil sampling location in *Eucalyptus grandis* plantation forest, Diyatalawa, Uva province, Sri Lanka.

Treatments were applied to air-dried aggregates selected from four depths using an automated programmable muffle furnace (Nabertherm GmbH muffle furnace, L-152S3-N3, Germany). All the treatments, including three levels of T_H, R_H, and E_D were carried out separately. During the heat treatments, aggregates were kept in crucibles of the same type and size and at the same sample height to avoid possible variations in heat transmission. After the heat treatments, aggregates were stored in the controlled environment chamber (65±5% RH at 28±2 °C) overnight to allow cooling.

Laboratory measurements

Molarity of an ethanol droplet (MED) test

The magnitude of SWR of aggregates was determined using the molarity of an ethanol droplet (MED) test (Poulenard et al., 2004), which is used to estimate soil-water contact angles. The measurements were conducted under laboratory conditions (65±5% RH at 28±2 °C). For the MED test, ethanol solutions with varying molarity (M) were prepared from 0 to 6 M in increments of 0.2 M. A drop of ethanol with 40±1 µL with increasing molarity was placed on an aggregate surface using a micropipette (Nichipet EX II, NPX2–100, Nichiriyo, Japan) until the drop completed the penetration into the aggregate within 10 s, and the relevant molarity was recorded (Roy and McGill, 2002). A separate aggregate taken from the relevant sample was used for each ethanol drop.

The molarity, x (mol L^{-1}) of the ethanol drop that penetrates the soil within 10 s can be converted to the 90° surface tension ($\gamma_{90°}$, mN m^{-1}), the surface tension of the ethanol solution at which the contact angle made on the soil is 90°, using Eq. (1). The Eq. 1 was derived to use at 25 °C using the data from the Japan Alcohol Association. The surface tension data were interpolated to obtain the data at 25 °C (Leelamanie et al., 2008; Letey et al., 2000; Roy and McGill, 2002).

$$\gamma_{90°} = 61.35 - 15.46 \ln (x + 0.5) \quad (1)$$

Then, the soil–water contact angle, θ, can be calculated by using Eq. (2) (Carrillo et al., 1999);

$$\cos \theta = (\gamma_{90°}/\gamma_w)^{0.5} - 1 \quad (2)$$

where γ_w represents the surface tension of the water–air interface (mN m^{-1}). This method is best suited for measuring the contact angles in the range of 90° to 109°. The lowest contact angle that can be determined using the MED test is 90°, where a water drop penetrates the soil within 10 seconds (Leelamanie et al., 2008; Roy and McGill, 2002). Considering this situation, the SWR levels of samples showing penetration times below 10 s with 0 molarity solution, i.e. water, (contact angle <90°), were differentiated using the water drop penetration time (WDPT) test.

Water drop penetration time (WDPT) test

The persistency of the SWR of aggregates was measured using the WDPT test (Chenu et al., 2000). To measure the WDPT, aggregates were placed on Petri dishes, and a drop of distilled water (40 ± 1 µL volume) was placed on the surface of the aggregates using the micropipette from about 1 cm height of the aggregate surface to avoid possible errors due to striking impact. The time required to disappear the water drop on aggregates visibly was recorded using a stopwatch. Measurements were taken in six replicates per sample. Water repellency of aggregates were classified based on the WDPT following the same categorization used for sieved soil samples as instant wetting (WDPT ≤ 1 s), non–repellent (WDPT = 1–5 s), slightly (WDPT = 5–60 s), strongly (WDPT = 60–600 s), severely (WDPT = 600–3600 s), and extremely water-repellent (WDPT = ≥3600 s) (Bisdom et al., 1993; Chenu et al., 2000; Leelamanie et al., 2008). All the measurements were conducted under laboratory conditions (65±5% RH at 28±2 °C).

Water stability of aggregates

The water stability of aggregates explains the ability of aggregates to withstand the forces of water. An index for the water stability of aggregates can be calculated as a proportionate fraction of the total weight of aggregates and can be expressed as a percentage of water stable aggregates (%WSA). The %WSA was determined in three replicates using a wet sieving apparatus (Eijkelkamp, Netherlands) (Piyaruwan and Leelamanie, 2020; Villamil et al., 2015). Prior to the wet sieving, the air-dried aggregates were pre-wetted for 3 h by keeping them on a moistened terry cloth. Approximately 4–5 g of aggregates were placed in single-sized sieves (250 µm) and the automated sieving was undertaken at 34 oscillations per minute with a 1.3 cm stroke through distilled water in weighing cans for 10 min. The soil suspensions in weighing cans were transferred into weighed evaporating dishes and evaporated, oven–dried, and weighed (M1). The same procedure was followed for the remaining aggregates in sieves using NaOH (2 g L^{-1}) in weighing cans instead of distilled water. The aggregates were scrubbed against the screen of sieves using a glass rod until all aggregates were disintegrated and only fragments of roots and sand particles were left on the sieve. The stable fraction of aggregates that passed through the sieves into weighing cans was evaporated, oven–dried, and weighed (M2). The %WSA was calculated gravimetrically using Eq. (3), where *M1* represents the weight of unstable aggregates (rapidly disrupted by the force of water) and *M2* represents the weight of water-stable aggregates.

$$\%WSA = \frac{M2}{M1+M2} \times 100\% \quad (3)$$

The percentage change in %WSA (ΔWSA%) aggregates as a result of heating was calculated using Eq. (4):

$$\Delta WSA\ \% = \frac{WSA_H - WSA_{NH}}{WSA_{NH}} \times 100\% \quad (4)$$

where WSA_H and WSA_{NH} are water stability of aggregates at heated and non–heated conditions, respectively.

Organic matter contents of aggregates

Organic matter contents of aggregates were measured using the loss on ignition method (Schumacher, 2002). Both heated and non-heated aggregates were kept in separate crucibles and heated at 105 °C for 24 h using a muffle furnace (Nabertherm GmbH muffle furnace L-152S3-N3). All the aggregates were stored in a desiccator for cooling before weighing. Then, the dry mass of the aggregates (m_1) was measured. After that, aggregates were ignited at 400 °C for 6 h and kept in a desiccator for cooling. The mass of the ignited aggregates (m_2) and the empty crucibles (m_3) was measured using an analytical balance. Finally, the organic matter contents of aggregates were calculated using Equation (5).

$$\text{Organic matter content (\%)} = \frac{m_1 - m_2}{m_1 - m_3} \times 100\% \quad (5)$$

The average of the three replicated was considered as the organic matter content of the particular sample.

Data analysis

Data were statistically analyzed using ANOVA and regression at a 95% confidence level ($p < 0.05$). All the measured parameters indicated in this paper are the mean values, and the error bars in the figures indicate the ± standard deviation.

RESULTS AND DISCUSSION

The MED values and the calculated contact angles are shown respectively in Table 2 and Figures 2–4. The SWR of the Eucalyptus plantation forest soils decreased with increasing depth. The surface layer (0–5 cm) showed the highest repellency (contact angle: ~103°±1), and the 15–20 cm layer showed non–repellent or sub-critically repellent conditions with the contact angle < 90° (WDPT = ≤1–2 s).

Figures 2, 3 and 4 present the SWR of aggregates, as measured by the contact angles, under three levels of R_H (200, 400, 800 °C h^{-1}) at 30, 60, and 120 min E_D for aggregates

Table 2. MED values of heated and non-heated aggregates.

		Molarity of an ethanol droplet that penetrates soil within 10 s (mol L^{-1})									
Heating rate (°C h^{-1})	Soil depth (cm)	30 °C	150 °C			200 °C			250 °C		
			30	60	120	30	60	120	30	60	120
non heated	0–5	2.4±0.4									
	5–10	1.5±0.3									
	10–15	0.1±0.2									
	15–20	0±0									
200	0–5		1.7±0.1	0.9±0.0	1.5±0.1	1.4±0.1	0.1±0.1	0±0	0±0	0±0	0±0
	5–10		0.9±0.1	0.0±0.1	0.1±0.1	0.4±0.1	0±0	0±0	0±0	0±0	0±0
	10–15		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0
	15–20		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0
400	0–5		1.8±0.1	1.0±0.1	1.8±0.1	1.4±0.1	0.7±0.1	0±0	0±0	0±0	0±0
	5–10		0.9±0.1	0.6±0.1	0.5±0.1	0.6±0.1	0±0	0±0	0±0	0±0	0±0
	10–15		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0
	15–20		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0
800	0–5		1.9±0.1	1.4±0.1	1.8±0.1	1.4±0.1	0.3±0.1	0.1±0.1	0±0	0±0	0±0
	5–10		1.6±0.1	0.4±0.1	0.6±0.1	0.7±0.1	0±0	0±0	0±0	0±0	0±0
	10–15		0±0	0±0	0.1±0.1	0±0	0±0	0±0	0±0	0±0	0±0
	15–20		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0

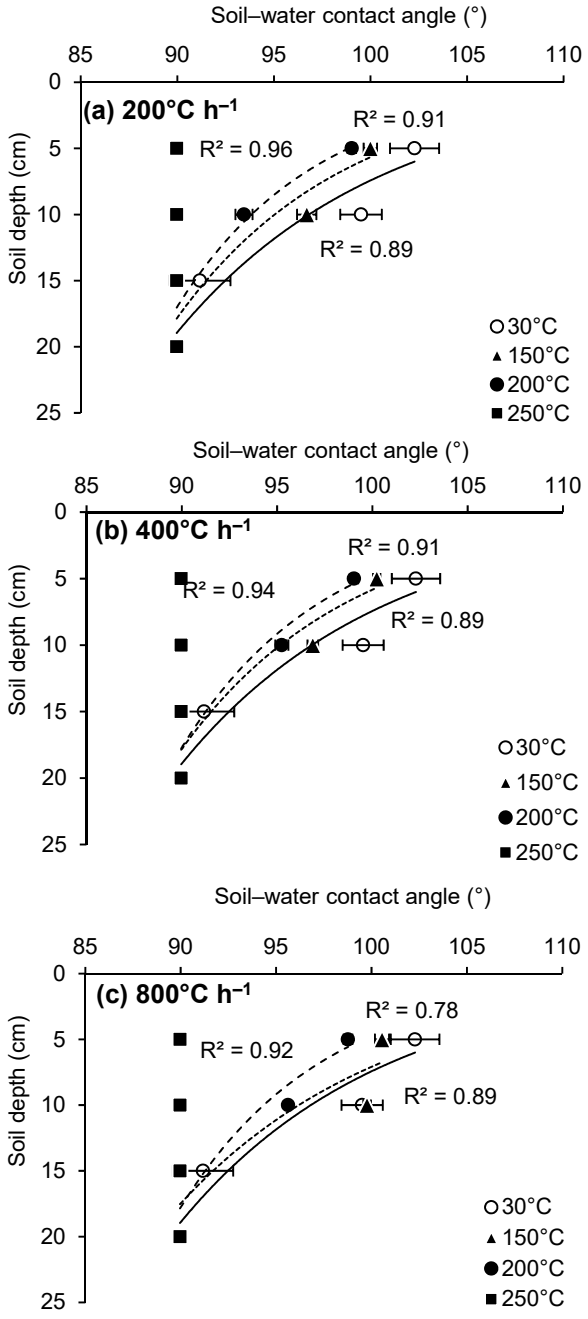

Fig. 2. Contact angles at 30 min duration of exposure under 200 °C h^{-1} (a), 400 °C h^{-1} (b), 800 °C h^{-1} (c) heating rates through the soil profile. Error bars indicate ± standard deviation.

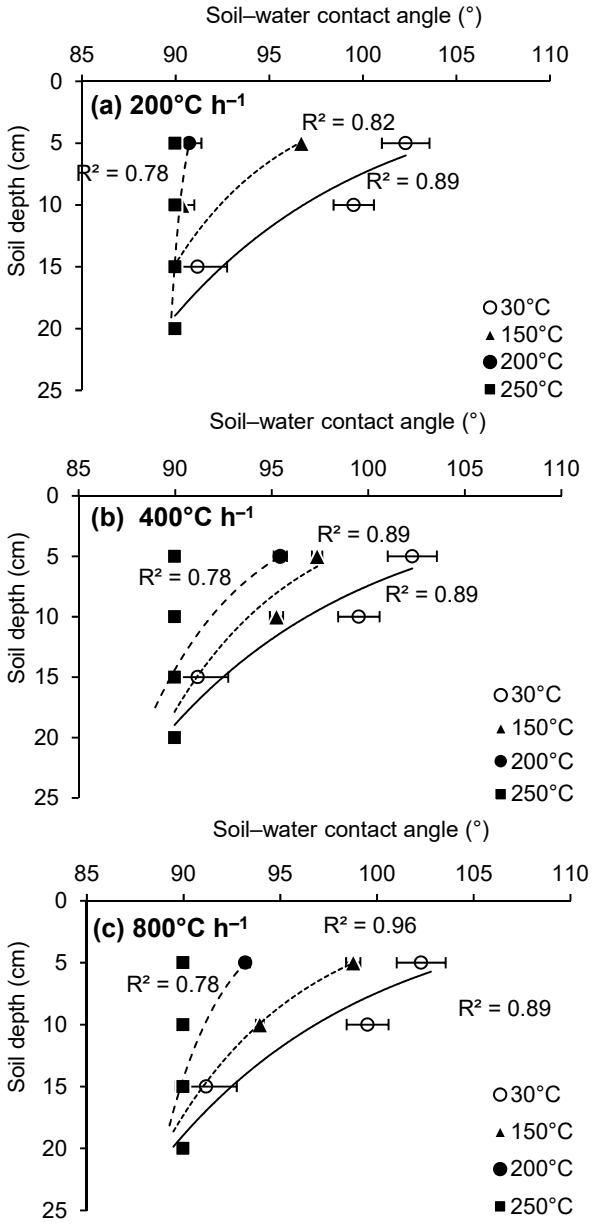

Fig. 3. Contact angles at 60 min duration of exposure under 200 °C h^{-1} (a), 400 °C h^{-1} (b), 800 °C h^{-1} (c) heating rates through the soil profile. Error bars indicate ± standard deviation.

obtained from different depths. Since the lowest contact angle measured using the MED test is 90°, where a water drop penetrates soil in 10 s, all values below that level were kept at 90° in the figures.

Under 30-min E_D, aggregates from 0–5 and 5–10 cm layers showed contact angles above 90° at 150 °C and 200 °C T_H**.** Results revealed that the R_H (200, 400, 800 °C h^{-1}) did not influence the behavior of SWR upon exposure to heat at the 30 min E_D (Figure 2 a, b, c). However, at 60 min E_D (Figure 3), the aggregates showed a difference in contact angles between three levels of R_H at 150 °C and 200 °C. The decrease in contact angle with increasing T_H under the slow R_H (200 °C h^{-1}) was greater (Figure 3a) compared with those at medium (400 °C h^{-1}, Figure 3b) and rapid R_H (800 °C h^{-1}, Figure 3c). The 0–5 cm layer aggregates showed contact angles >90° at 150 and 200 °C T_H. The 5–10 cm layer aggregates showed contact angles >90° at 150 °C under 400 and 800 °C h^{-1} R_H (Figure 3 b, c). The aggregates from 10–15 and 15–20 cm layers showed contact angles below 90° at all heat treatments. At 120 min E_D (Figure 4), contact angles of aggregates were <90° at 200 °C, except for the aggregates from the surface layer (0–5 cm) at the rapid R_H (800 °C h^{-1}, Figure 4c). The contact angles of 0–5 and 5–10 cm layer showed values above 90° at 150 °C T_H. At 250 °C, all samples showed instant wetting or non-repellent conditions (Table 3) with contact angles below 90° irrespective of the E_D and R_H (Figures 2, 3, and 4). The SWR of aggregates in the deeper soil

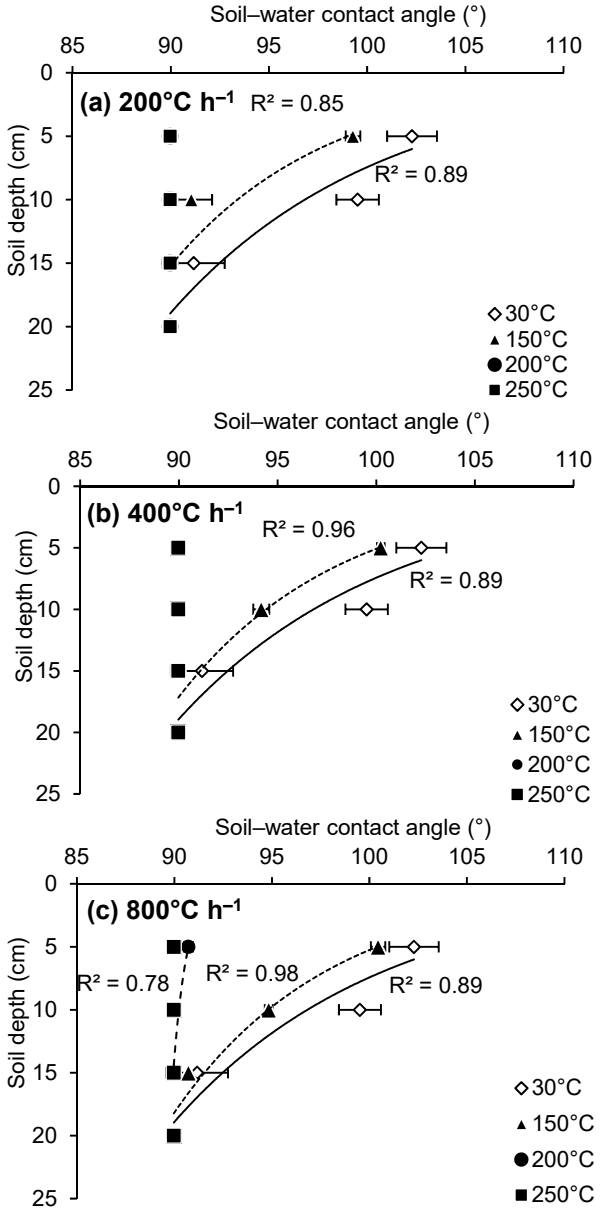

Fig. 4. Contact angles at 120 min duration of exposure under 200 °C h^{-1} (a), 400 °C h^{-1} (b), 800 °C h^{-1} (c) heating rates through the soil profile. Error bars indicate ± standard deviation.

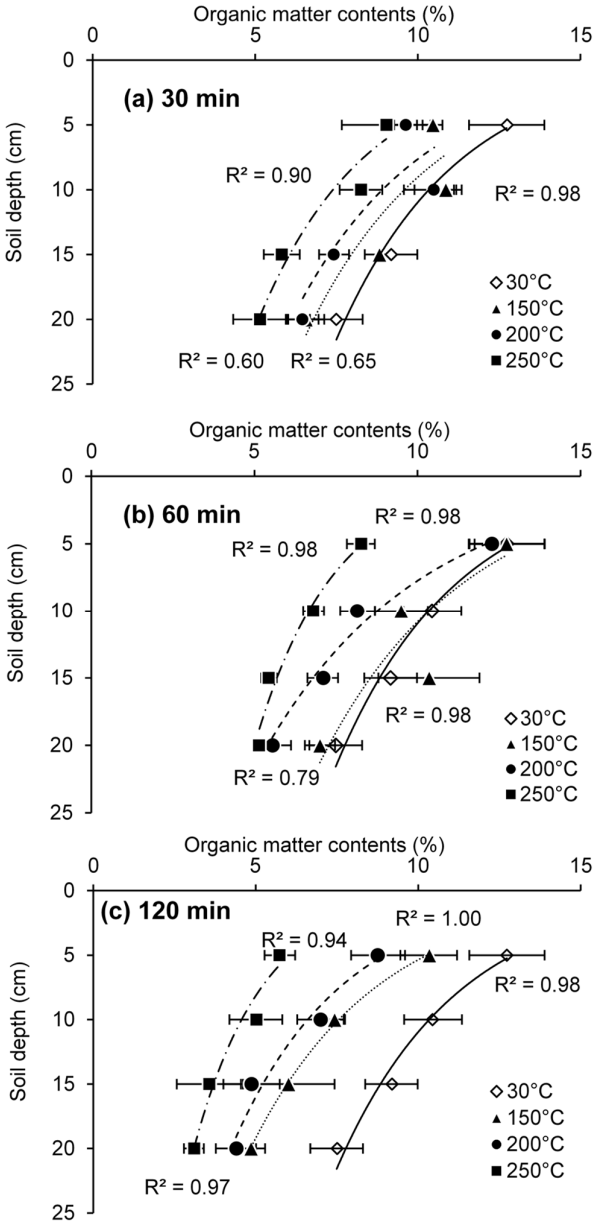

Fig. 5. Organic matter contents of soil aggregates along the soil profile after heating to 150 °C, 200 °C, 250 °C temperatures for (a) 30, (b) 60, and (c) 120 min durations of exposure under medium rate heating (400 °C h^{-1}). Error bars indicate ± standard deviation.

layers (10–15 and 15–20 cm) showed contact angles below 90° with the lowest T_H used in the experiment (150 °C, 30 min), irrespective of the R_H and E_D. Following the similar trend of contact angles, the WDPT also showed lower values at slow R_H compared with those at medium and R_H (Table 3).

The heat generated during a fire alters the SWR, where the heat may induce, decrease or eliminate water-repellent conditions in soils (Caltabellotta et al., 2022; Doerr et al., 2004, 2005; Negri et al., 2021; Plaza-Álvarez et al., 2018; Zavala et al., 2010). In this study, however, the SWR of aggregates did not show any increase with heating treatments (R_H, T_H, E_D). It was clear that the R_H did not significantly influence the contact angle, although the contact angles of aggregates taken from 0–5 and 5–10 cm layers indicated that the slow R_H tends to lower the SWR more compared with rapid R_H (Table 3). SWR showed a general declining trend with increasing T_H and E_D. Depending on the initial SWR levels, repellency showed elimination (samples became non-repellent) with increasing T_H and E_D. These results are comparable with Novák et al. (2009), who reported a decline in SWR with increasing temperature in Meadow and Pine Forest soils. Dao et al. (2022) and Doerr et al. (2005) also reported an increase in SWR with increasing temperature up to about 150 °C, following a decline in SWR with heating beyond 150 °C.

The R_H in this study describes the speed of rising temperature to reach the relevant T_H, and indirectly, the time taken to reach the selected T_H (represents a slow and rapid increase in temperatures). Accordingly, slow, medium, and rapid R_H (200, 400, and 800 °C h^{-1}) respectively required 45, 22, and 11 min, to reach 150 °C, 60, 30, and 15 min, to reach 200 °C, and 75, 37, and 18 min, to reach 250 °C. Therefore, the total time taken for

the overall heating process changes with the R_H. A fast-moving fire releases relatively low energy compared to a slow-moving fire (Neary et al., 1999). Therefore, slow heating would provide sufficient time and energy for the gradual volatilization of water-repellent organic materials. However, it was not possible to notice any influence of the R_H on SWR at the highest T_H (250 °C) and lowest E_D (30 min) (Figure 2 and Table 3). Elimination of repellency, or the extent of decrease in repellency level, was found to be dependent on the E_D and T_H, showing that even the shortest E_D eliminated SWR at the highest level of T_H, while lower T_H required longer E_D for similar results. These results are in line with Doerr et al. (2004) who reported that the elimination temperature for repellency is dependent on heating duration in Australian eucalypt forest soils.

The organic matter contents of soil aggregates heated to different T_H under medium-rate R_H (400 °C h^{-1}) are indicated in Figure 5. Only medium rate R_H was used because there was no significant difference in organic matter contents between the three R_H levels**.** In general, the organic matter contents of aggregates decreased with increasing depth, T_H, and E_D. Soil organic matter is crucial for the occurrence of SWR under both burned and unburned conditions (Atanassova and Doerr, 2010; Dao et al., 2022). In our study, we evaluated the relationship between soil organic matter content and SWR by examining contact angles (Figure 6). Our findings indicate a significant ($p<0.05$) moderate positive linear correlation ($R^2 = 0.65$) between organic matter content and contact angle. Therefore, the decrease or absence of SWR observed after heating events may be attributed to the reduction in organic matter content caused by organic matter combustion.

Figure 7 shows the percentage change (increase) in water stability of aggregates (ΔWSA%) under medium-rate heating (400 °C h^{-1}). Since ΔWSA% did not show any significant difference between values observed for three R_H, the figure presents only the data obtained at the medium R_H. The ΔWSA% was higher in aggregates obtained from the deeper soil layers, indicating an increasing trend for all E_D except for samples from 15 to 20 cm layer at 30 and 120 min E_D, which showed a decrease.

Although organic matter content, which is one of the primary stabilizing agents of aggregates (Vogelmann et al., 2013),

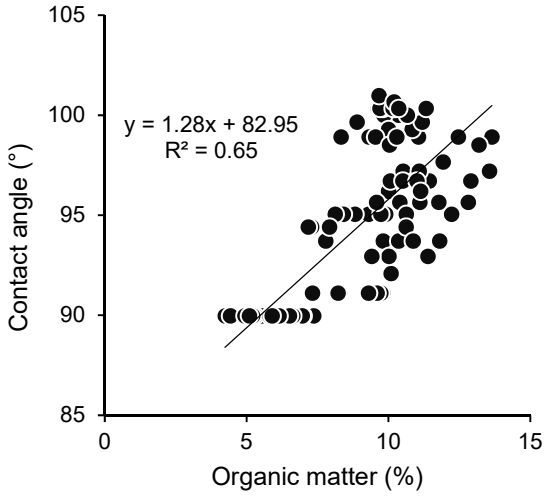

Fig. 6. Relation of organic matter content to the contact angle. The standard error of the slope and the intercept are 0.05 and 0.40, respectively.

decreased with heating in the present study, the water stability of aggregates increased. Reports are available to show that the organic matter content does not primarily affect the stability of aggregates in burned soil (Guerrero et al., 2001). At the initial level, the %WSA of the soil decreased with increasing depth showing comparable results with the findings of Piyaruwan and Leelamanie (2020). However, this trend altered after heating. The aggregates from lower depths with a low level of repellency showed higher ΔWSA% (Figure 7) compared with the aggregates from upper layers, which initially showed a high level of repellency. These results are in agreement with the previously reported results in the literature (Arcenegui et al., 2008; García-Corona et al., 2004; Terefe et al., 2008). Terefe et al. (2008) reported that heating aggregates to temperatures below 300 °C can increase their water stability, while García-Corona et al. (2004) reported an increase in the stability of aggregates that are exposed to heat at 170–220 °C.

Table 3. Water drop penetration times (WDPT) of aggregates.

WDPT (s)											
Heating rate	Soil depth	30 °C	150 °C			200 °C			250 °C		
(°C h^{-1})	cm		30	60	120	30	60	120	30	60	120
non heated	0–5	7200±0									
	5–10	510±17									
	10–15	15±3									
	15–20	2±1									
200	0–5		309±98	112±10	261±36	83±49	14±4	4±1	2±4	0±0	0±0
	5–10		123±43	11±2	19±3	19±10	3±2	0±0	0±0	0±0	0±0
	10–15		2±1	2±1	7±2	0±0	0±0	0±0	0±0	0±0	0±0
	15–20		0±1	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0
400	0–5		429±170	143±14	361±10	169±51	89±19	22±3	1±1	0±0	0±0
	5–10		185±30	85±16	43±6	80±20	4±2	0±0	0±0	0±0	0±0
	10–15		13±3	6±3	6±2	0±0	0±0	0±0	0±0	0±0	0±0
	15–20		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0
800	0–5		759±250	96±34	704±69	297±134	35±6	12±3	1±1	0±0	0±0
	5–10		592±185	32±11	52±5	160±99	5±2	0±0	0±0	0±0	0±0
	10–15		7±5	0±0	13±4	6±3	0±0	0±0	0±0	0±0	0±0
	15–20		0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0	0±0

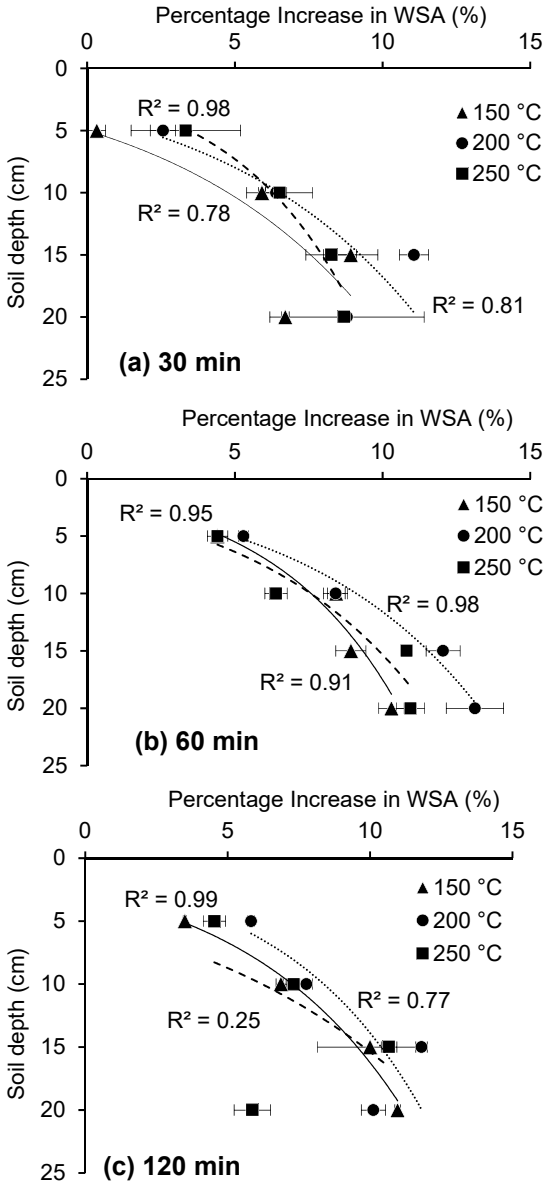

Fig. 7. Percentage increase in water–stable aggregates (WSA) along the soil profile after heating to 150 °C, 200 °C, 250 °C temperatures for (a) 30, (b) 60, and (c) 120 min durations of exposure under medium rate heating (400 °C h^{-1}). Error bars indicate ± standard deviation.

Most previous studies indicate that an increase in SWR is the cause of the improvement of the stability of aggregates. The rapid build-up of air pressure inside the aggregates owing to the rapid water entry into intra-aggregate pores leads to the disruption of aggregates. Since SWR hinders the rapid water entry into aggregates and pressure build-up in intra-aggregate pores, water-repellant soils tend to exhibit comparatively high aggregate stability (Arcenegui et al., 2008; Blanco-Canqui and Lal, 2009). Interestingly, in the present study, the %WSA increased with heating irrespective of the decreasing SWR, specifically in lower soil depths with low initial SWR (Figure 7).

Observations in our study lead to the possibility of the occurrence of some phenomena during heating, that prevented water entry into intra-aggregate pores. According to capillary principles, the soil-water contact angle in pore walls has to be below 90° for water to enter into pores. The presence of water-repellent compounds on pore walls causes the soil-water contact angle to be higher than 90°. Heating to temperatures between 100 and 200 °C leads to the volatilization of water–repellant organic materials present in aggregates (González-Pérez et al., 2004 and references therein). Temperature gradients might develop during the heating processes, facilitating volatilized organic materials to move and condense in comparatively cooler areas (DeBano, 2000). The outer surface of the aggregates can reach the temperature levels quickly, resulting in a loss of the water–repellent materials making the surface of aggregates less or non-repellent. However, the volatilized organic materials might move inside the intra-aggregate pores following temperature gradients and condense (Terefe et al., 2008 and references therein) forming water-repellent pore walls, preventing or retarding the entry of water preventing rapid pressure buildup within aggregates.

However, some aggregates (from 10–15 and 15–20 cm depths) that showed instant wetting after heating exhibited increased stability at 150 and 200 °C (Figure 7). These aggregates did not show high levels of SWR at initial conditions (before heating) as well. Therefore, the presence of highly repellent material to be volatilized or deposited on or within aggregates seemed unlikely. Still, the increased %WSA indicates hindrance of water entry into intra-aggregate pores and decreased pressure build-up within aggregates avoiding disruption. As reported by Atanassova and Doerr (2010), there are conformational changes that might occur in amphiphilic organic compounds present in soils (Ex: fatty acids, polar compounds, small molecular weight aromatic acids, phenolic compounds) due to physical stresses such as wetting and drying. Such compounds with their polar groups originally oriented outwards into soil pore spaces exhibit non-repellent conditions. Conformational changes in these molecules may alter the orientation of organic compounds leading to attaching polar groups to the mineral surfaces, exposing hydrophobic ends into the soil pore space, and resulting in water-repellent pore walls. A similar phenomenon that might occur inside the aggregates can be considered as one of the possible reasons for the aggregates from 10–15 and 15–20 cm depths to exhibit high stability. Furthermore, some reports explain the increase in aggregate stability of burned soils as a result of soil gel dehydration at higher temperature levels above 170 °C and 220 °C (Giovannini et al., 1990; Guerrero et al., 2001; Soto et al., 1991). In the present study, this kind of soil gel dehydration can also be considered as another possible cause for the increased stability of aggregates exposed to heating. The exact reasoning would require further investigations.

CONCLUSIONS

In this laboratory experiment, the effects of three dynamics of heating (T_H, R_H, E_D) on the SWR, and stability of aggregates were investigated using a water-repellent Eucalyptus Forest soil. According to the findings, SWR showed a general trend of declining with increasing temperature and the duration of exposure. Complete elimination of SWR occurred with increasing T_H and E_D, depending on the initial level of SWR. The R_H showed the least influence on all measured parameters.

Although water repellency is a factor that causes high stability of aggregates, results revealed increased stability of aggregates irrespective of the decreasing SWR, including those in lower soil depths with low initial SWR. Deposition of volatilized organic material in intra-aggregate pores or molecular conformational and orientational changes in organic compounds that create water-repellent pore walls can be considered as possible reasons

for the resistance to water entry, leading to the high stability of aggregates.

Although laboratory experiments would not precisely represent the behaviors of aggregates under natural fire events, this study represents the heating dynamics that might occur in natural fire events. Further investigations on the heat-induced changes in organic compounds at molecular levels would be necessary for the affirmation of the suggested theories.

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Conflicts of interest. As the authors of the manuscript, herewith we confirm that this study did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors, and that there are no conflicts of interest in any manner.

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