

Reburning pyrogenic organic matter: a laboratory method for dosing dynamic heat fluxes from above

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ABSTRACT

Background. Pyrogenic organic matter (PyOM) represents a relatively persistent component of soil carbon stocks. Although subsequent fires have the potential to combust or alter preexisting PyOM stocks, simulating soil heating faces important methodological constraints. In particular, methods for estimating the effects of subsequent fire on preexisting PyOM in soil have important limitations. **Aims.** We aimed to design a laboratory method to effectively simulate soil heating from above, to investigate the impacts of subsequent fires on PyOM at different soil depths while addressing key limitations of previous methods. **Methods.** Jack pine (*Pinus banksiana* Lamb.) log burns were used to parameterise realistic heat flux profiles. Using a cone calorimeter, these profiles were applied to buried jack pine PyOM to simulate variable reburn fire intensities. **Key results.** In general, higher heat fluxes and shallower depths led to more mass loss of PyOM. **Conclusions.** We offer a method to simulate specific soil heating profiles. Conditions that result in higher temperatures (higher heat fluxes and shallower depths) are likely to lead to more loss of PyOM in subsequent fires. **Implications.** The method could simulate different fire scenarios to represent spatial variability within a given fire event, or to study the effects of fire on different types of biomass, or organisms such as microbes.

Keywords: cone calorimeter, depth, fire, heat flux, jack pine, log burn, pyrogenic organic matter, soil, soil heating.

Introduction

Fire effects on preexisting pyrogenic organic matter

Pyrogenic organic matter (PyOM), produced from incomplete combustion of organic matter (OM) (Bird *et al.* 2015), represents a persistent (Spokas 2010) and, in many cases, large component of soil organic carbon (C) (Czimczik and Masiello 2007; Boot *et al.* 2014; Reisser *et al.* 2016). PyOM is generally a larger fraction of total soil C in ecosystems with more frequent fires (Reisser *et al.* 2016) and those for which fuel-reduction efforts include the production and application of PyOM for soil health or C management purposes (Franco *et al.* 2024). However, the burns that produce PyOM rarely occur in isolation. Over a sufficiently long timescale, almost all fires could effectively be classified as reburns, and over human timescales, fires occur frequently in many different anthropogenic and natural contexts. Whether due to naturally high-frequency fire regimes (Holden *et al.* 2010; Prichard *et al.* 2017), decreasing fire return intervals (Westerling *et al.* 2006; Mansoor *et al.* 2022), or repeated prescribed fires used to maintain or restore landscapes (Prichard *et al.* 2017; Lutz *et al.* 2020; Saberi and Harvey 2023), subsequent fires are an important part of understanding the context in which a given fire occurs and its effects on PyOM.

What happens to PyOM in a subsequent fire? Does it stay in the soil, or is it largely combusted? Does it become more persistent, or more readily degradable? Important methodological constraints limit our understanding of the effects of subsequent fires on PyOM stocks. Of the relatively few previous studies that have examined the impact of fires on PyOM, most of them are field studies. For example, the study conducted by Santín *et al.* represents one of the earliest field and laboratory experiments on the net

effect of fire on pre-existing PyOM (Santín *et al.* 2013). This study provides direct evidence that fire is an important driver for PyOM loss. Past relevant studies have generally concluded that in natural ecosystems, fire can add, consume, and transform pre-existing PyOM (Saiz *et al.* 2015; Tinkham *et al.* 2016; Doerr *et al.* 2018; Bartoli *et al.* 2021). In order to investigate and understand these effects, we must find appropriate ways to simulate soil heating by fires.

Simulating soil heating

Soil heating can occur for many reasons, ranging from natural daily and seasonal fluctuations, to management practices like soil solarisation (Al-Shammmary *et al.* 2020) or thermal remediation of contaminated soils (O'Brien *et al.* 2018), to the effects of fires. When field measurements are not possible or not appropriate, options for accurately simulating soil heating in a laboratory setting can be limited. An oven (e.g. Acea and Carballas 1999), muffle furnace (e.g. Bahureksa *et al.* 2022; Myers-Pigg *et al.* 2024), or hot water bath (e.g. Bollen 1969) can all target specific soil temperatures, but they generally do not produce depth-resolved effects and, instead, heat the soil from all sides (Brucker *et al.* 2022). Heating lamps can offer overhead, depth-resolved warming, but may be difficult to tune precisely and have relatively low maximum temperatures (Klopatek *et al.* 1988). For fires, where target temperatures at the soil surface can be hundreds of degrees Celsius, none of these options is ideal. Simulating fires by burning a known mass of fuel at the surface in 'pyrocosms' has produced replicable heating profiles with depth (Bruns *et al.* 2020). Open-air burn tables/plates can also simulate fires on a small scale and allow tracking of fire properties over the duration of the burn (Bartoli *et al.* 2021; Myers-Pigg *et al.* 2024). However, in studies focused on chemical effects, the introduction of material from the fuel can confound subsequent chemical analyses. Furthermore, it can be difficult to fine-tune the fuel's combustion to represent different types of heating profiles that are representative of realistic field conditions. Given these constraints, Brucker *et al.* (2022) have called for improved methods for soil heating.

Improving simulations of heat effects on PyOM

For investigations of the effects of repeated fires on PyOM, constraints in typical methodological designs for soil heating can be grouped into four key areas: (1) incomplete representation of PyOM, (2) overlooking the PyOM in mineral soil, (3) contamination (i.e. introducing new PyOM) from fuel, and (4) difficulties in quantifying fire intensity, therefore limiting predictive power or extrapolation beyond the specific system.

Incomplete representation of PyOM

It is extremely difficult to completely collect or quantify all PyOM in a field setting. Prior studies have found that the particle size and shape of PyOM do not significantly affect

mass loss through combustion, within the range of particle sizes studied (Santín *et al.* 2013; Tinkham *et al.* 2016; Bartoli *et al.* 2021). However, these findings also reflect the fact that smaller size fractions of PyOM were not collected for the analyses. Small-sized PyOM, including soot and aerosol 'black carbon,' is often highly persistent but generally too mobile or small to be effectively collected and analysed (Ansley *et al.* 2006; Matosziuk *et al.* 2019). Consequently, studies including or focusing on the smallest size fractions of PyOM are limited, despite the potential implications for C cycling.

Overlooking PyOM buried in mineral soil

A second challenge in studying PyOM after subsequent fires is the difficulty in separating or distinguishing it from other soil OM. One group of methods for isolating PyOM is typically destructive and relies on the general principle of removing chemically labile OM and classifying the rest as PyOM (Zimmerman and Mitra 2017). Thermal oxidation, such as the CTO375 method (Gustafsson *et al.* 1996; Hatten *et al.* 2008) and acid oxidation, such as the benzenepolycarboxylic acid method (Brodowski *et al.* 2005; Matosziuk *et al.* 2020) and hydrogen pyrolysis (Ascough *et al.* 2009), are some of the most commonly used approaches. These infer PyOM stocks from C compounds with high chemical recalcitrance. However, they are not fully accurate because the highly degradable portion of PyOM is excluded ('false negatives'), and some of the persistent C may not have been produced from heating ('false positives'). Typical non-destructive methods, such as mid-infrared spectroscopy, also suffer from potential false positives and false negatives (Zimmerman and Mitra 2017) and preclude direct chemical and biological analyses of the PyOM on its own.

As a result of these methodological limitations, most studies have focused on PyOM on the surface or in the organic horizon, where it can be visually separated from unburned OM, and not in the mineral soil. However, over time, surface PyOM moves from the organic horizon to mineral soil (Tinkham *et al.* 2016; Matosziuk *et al.* 2020). This vertical movement of PyOM can occur through processes such as bioturbation or translocation with water, while PyOM can also be produced at depth through the charring of roots and buried biomass *in situ* (Hobley 2019; Soucémariadin *et al.* 2019). Previous studies of subsequent fires have suggested that PyOM residing in deeper soil horizons can be protected from heat and combustion due to soil insulation (Santín *et al.* 2013; Saiz *et al.* 2015; Doerr *et al.* 2018; Bartoli *et al.* 2021), meaning it is particularly important to understand the interactions between burial depth and PyOM loss or alteration in subsequent fires. However, there is a lack of comparative experiments to validate these expectations, with most studies focusing on PyOM either on the surface or within the litter layer, not in the mineral soil.

Contamination from fuel

The use of a surface fuel is a straightforward method for simulating fire effects on soils (Bruns *et al.* 2020). However,

this method can contaminate the preexisting PyOM in the system due to the addition of new PyOM from the fuel, making it challenging to analyse the net effect of fire on the PyOM. Some studies have attempted to address this issue by wrapping the PyOM sample in a metal mesh bag to separate it from the fuel (Santín *et al.* 2013; Bartoli *et al.* 2021). However, metal can alter the heat transfer to the sample, and small particles can still leak out from or into the mesh bag.

Quantifying and controlling fire intensity

Fire intensity is defined as the ‘energy output’ of a fire and is not explicitly linked to any effects caused by fire (Keeley 2009). However, quantifying fire intensity is difficult, particularly in natural settings, and controlling it using other fire simulation methods is also challenging. In laboratory settings, simulated fires in the past studies are not always tied to real-world parameters, which limits their broader applicability (Brucker *et al.* 2022).

Methodological approach and hypotheses

We aimed to develop a method for simulating the effects of subsequent fire on PyOM that effectively addresses each of the limitations discussed above. Our approach simulates soil heating in a sand matrix using a cone calorimeter with high-resolution incremental controls and is highly versatile, with potential applications across a wide range of soil heating scenarios. We applied this method to study the effects of burial depth and heat flux on PyOM mass loss. We hypothesised that higher heat flux and shallower depths would have more PyOM mass losses. At the same heat flux, we predicted that PyOM on the surface would be subject to more mass loss than PyOM at both 1 and 5 cm depths. At the same depth, we predicted that high heat-flux (High HF) fire would consume more PyOM than low heat-flux (Low HF) fire.

Methods

Ecosystem, site description, and experimental overview

We designed our burn experiments to generally reflect the ecosystem of the jack pine (*Pinus banksiana* Lamb.) barrens in Wisconsin, where the soil is characterised as coarse sand with low nutrients, low water holding capacity, and prone to drought (Radeloff *et al.* 1999, 2004). Jack pine barrens in Wisconsin encompass a range of fire regimes, with the shortest fire return intervals being about 3–7 years (Radeloff *et al.* 2004). For this experiment, we used two jack pine trees that were cut down from the Wilson State Forest Nursery (43.1461°N, 90.6950°W) and the Hancock Agricultural Research Station (44.1227°N, 89.5307°W) in Wisconsin.

Briefly, to simulate the effects of subsequent fire on PyOM, we pyrolysed jack pine wood to produce PyOM, and then

burned the resulting PyOM under a cone calorimeter at the Forest Products Laboratory of the USDA Forest Service in a sand matrix. We designed the fire to be representative of logs burning on the soil surface in a jack pine stand, including a higher and lower fire intensity. We directly quantified the heat flux transferred downwards into the soil in this system, and chose this approach to represent a scenario where the greatest heat transfer to soil might be likely to occur, thereby capturing the upper bounds of fire effects in this system.

Production of PyOM

The PyOM for this study was produced from ground jack pine wood (<2 mm) at a highest treatment temperature of 350°C in a muffle furnace (Thermo Fisher Scientific 1100°C Box Furnace BF51800 Series; Güereña *et al.* 2015; Zeba *et al.* 2022) modified to operate under an argon gas atmosphere; 350°C is within the temperature range of a typical low-intensity forest fire (Santín *et al.* 2016b). We increased the temperature from 25 to 250°C at 5°C min⁻¹, then increased it to 350°C at 5°C min⁻¹ with the rotor on (to stir the PyOM in the chamber) and sustained it at 350°C for another 30 min with the rotor on. After that, we stopped the rotor and water was circulated outside the pyrolysis chamber to rapidly cool the PyOM. The PyOM was collected once it reached room temperature. The PyOM yield was ~35% of the unburned jack pine wood by dry mass. This PyOM thus represents PyOM produced in the first fire, while the fire simulated in the experimental burns with the calorimeter is considered to be the subsequent fire for the purposes of this experiment.

Determining fire intensity via heat transfer sensing in log burns

To ensure that the heat treatments were ecologically realistic, we designed experimental log burns to determine the heat flux dose for the High and LowHF treatments. The logs used in this experiment were stored in a drying room at 32°C and 30% relative humidity for 6 months. The logs were then oven dried at 105°C for >48 h before the burn. A sand bed was prepared and levelled for the burn with three embedded water-cooled heat flux sensors. Two were Hukseflux combination Gardon and Schmidt-Boelter heat flux sensors with a measurement range of 0–200 kW m⁻² (model SBG01-200). The third was a Medtherm 64 series Schmidt-Boelter heat flux sensor with a range of 0–114 kW m⁻² (model 64-10SB-18). All the sensors were calibrated under the cone calorimeter before the deployment. The tops of the sensors were at the same level as the surface of the sand bed, and they were linearly aligned in the centre of the sand bed (Fig. 1b). The first log was suspended 12 cm above four natural gas Bunsen burners for 10 min, after having a small groove cut by hand to increase surface area for combustion (Fig. 1a). The burning log was then placed parallel to the second log with the groove facing the heat flux sensors and angled such that one end was elevated approximately 10 cm above the sand to

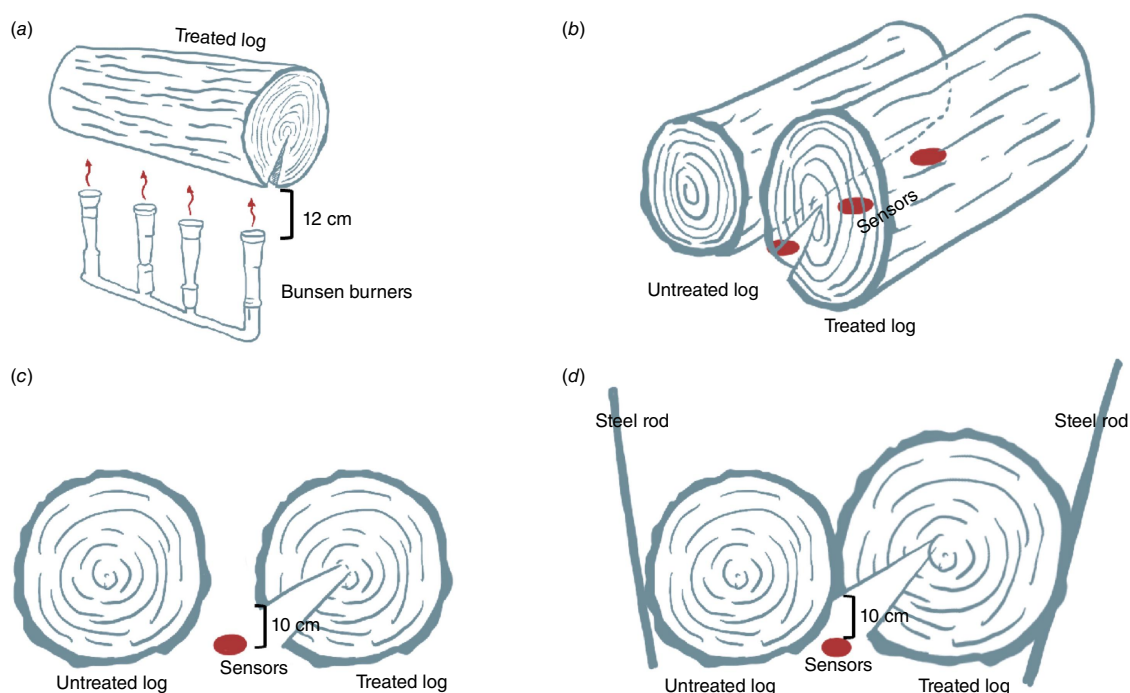


Fig. 1. Log burn preparation and setup (corresponding photos displayed in Supplemental Fig. S1). (a) First log on burners; (b) log and sensor placement; (c) the two logs' relative sizes and location for the Low HF profile (cross section displayed; lengths, 610 mm for both; diameters, left log 165 mm and right log 155 mm; masses, left log 5717 g and right log 5052 g); and (d) the two logs' relative sizes and location for the High HF profile (cross section displayed; lengths, 610 mm for both; diameters, left log 125 mm and right log 180 mm; masses, left log 3943 g and right log 8500 g).

promote buoyant gas flows (Fig. 1b–d). The two logs were situated on the sand bed such that the three heat flux sensors were in the middle of the two logs and ran parallel to the length of the logs (Fig. 1a, c, d). The principle for this approach was to sustain a controlled, laboratory-scale burn in order to obtain downward heat flux profiles from the paired logs that would then be input into the calorimeter for burning the PyOM samples.

We used the heat flux data of two two-log experimental burns to inform two fire scenarios – High HF and Low HF – which represented the upper and lower heat flux profiles out of all the log-burning trials performed. Both heat flux profiles were determined from the three sensors between the two logs. For the Low HF profile, the two logs were of similar size and were not stabilised in place (Fig. 1c; lengths, 610 mm for both; diameters, 165 and 155 mm; masses, 5717 and 5052 g). As the biomass was consumed by fire, the distance between the logs increased and measured heat flux declined. For the High HF profile, we wanted to model higher fuel connectivity and a higher fuel load. Thus, we maintained a relatively constant distance between the two logs with four steel rods that kept the two logs in place, and the ignited log was larger than the second log (Fig. 1d; lengths, 610 mm for both; diameters, 125 and 180 mm; masses, 3943 and 8500 g).

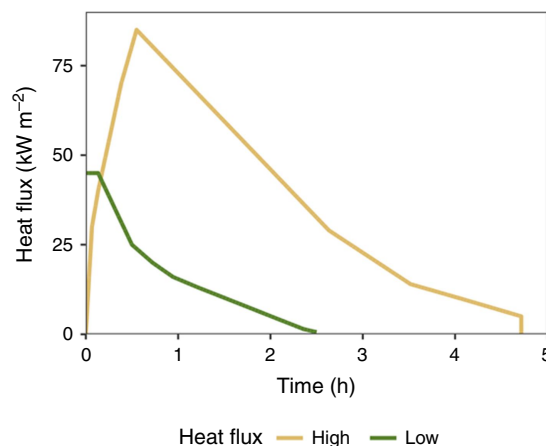


Fig. 2. High and low heat flux profiles delivered to the samples. Heat flux profiles were based on data from log burns.

The High HF profile, representing the ‘high-intensity fire’, was then replicated and monitored in the cone calorimeter using LabVIEW (National Instruments 2013) with a peak heat flux at 85 kW m^{-2} and a duration of 4.72 h. The Low HF profile, representing the ‘low-intensity fire’, was replicated with a peak heat flux at 45 kW m^{-2} and a duration of 2.5 h (Fig. 2).

Sample treatments and the burning matrix

To replicate the natural integration of PyOM into mineral soil in a field setting, for each sample, 1 g of PyOM was mixed with 8 g of sand before burying it in the pyrocosm's sand matrix. Three different depths of PyOM (surface and 1 and 5 cm) and three fire treatments (High HF, Low HF, and Control) were combined in a full-factorial design, for nine treatments in total. For each treatment, five samples of PyOM were placed at the appropriate depth in a sand matrix in a pyrocosm and exposed to the selected heat treatment (Fig. 3). Thus, a total of nine pyrocosms were prepared for this investigation.

Pool filter sand (0.45–0.55 mm, AquaQuartz) was used as the matrix for all treatments, representing sandy soils typical of jack pine stands while ensuring a C- and nutrient-free system. To eliminate any traces of C, the sand was treated in advance by heating it at 500°C for 3 h in a muffle furnace ('ashed'). Then it was washed with Milli-Q water to flush out any soluble chemicals ('washed'), oven dried at 100°C for more than 48 h to eliminate moisture, and stored in autoclaved media bottles.

The ashed and washed sand was used to fill a steel container (the 'pyrocosm'). The container was modified from a stainless steel beaker (McMaster-Carr stainless steel beaker with handle, 2850 mL capacity), with an internal diameter of 152.4 mm, internal depth of 90 mm, and a total volume of 1641.7 cm³ (Supplementary Fig. S2). The sand matrix had a mean bulk density of 1.54 g cm⁻³.

Five PyOM replicates for each fire treatment were placed in the sand matrix using a custom-designed 3D-printed PLA (polylactic acid filament) sample placer (modelled with Tinkercad; printed with Ultimaker S5, University of Wisconsin-Madison Makerspace), which is a flat plate embedded with five identical open-ended tubes (Supplementary Fig. S3). Each tube has a diameter of 30 mm and a depth of 16 mm (with volume of ~11.3 cm³). The centre of each tube is equidistant (45 mm) to the centre of the plate and the same distance (52.9 mm) from the adjacent tubes (Supplementary Fig. S3). The sample placer ensured that the samples were placed at the same level in the

pyrocosm and same distance from each other at each treatment. To place the samples at different depths for each treatment, we levelled the sand and pressed the sample placer into the sand. Then we scooped out the sand from each tube and filled the hollows with the samples, and gently removed the sample placer. We mixed the 1 g of PyOM with 8 g of sand before burying it. After placing the samples, for the 1- and 5-cm depths, we added sand to bury the samples and levelled the top of the matrix at 10 mm from the top of the container. Thermocouples made from 30-gauge type K wire (Omega Engineering, GG-K-30-SLE) were placed above and below each sample (Fig. 3a–f).

Everything in the pyrocosm, including the steel container, ring for stabilising thermocouples, and thermocouples, were weighed individually and all together before and after the burn to determine total mass loss of the samples and any extra mass loss of the appliances for each treatment. We also ran a 'blank trial' for both heat flux profiles with only sand to measure how much sand was volatilised during the burn. The mass loss of the whole pyrocosm with sand and PyOM was measured for each treatment. Mass loss fraction and mass remaining (%) were calculated with Eqns 1 and 2:

$$\text{Mass Loss Fraction} = \frac{\text{Mass}_{\text{before}} - \text{Mass}_{\text{after}}}{\text{Mass}_{\text{before}}} \quad (1)$$

$$\text{Mass Remaining (\%)} = (1 - \text{Mass Loss Fraction}) \times 100\% \quad (2)$$

Before placing the matrix under the cone calorimeter, an insulation sheet was wired around the container (Fig. 4) to prevent the thermocouples from being burned and to help ensure energy only enters or leaves the pyrocosm through the top surface. The pyrocosm was dosed using the pre-set heat flux profiles as described above. The temperature data for each thermocouple were recorded using LabVIEW (National Instruments 2013).

After each burn, the pyrocosm with the sand matrix was carefully removed from the calorimeter and set in a hood to

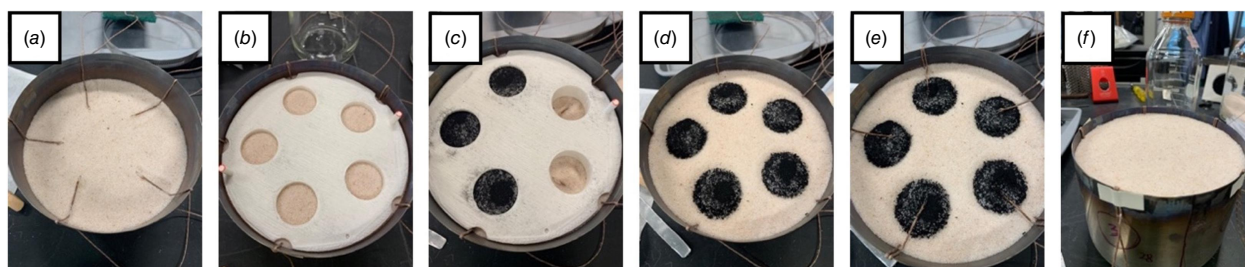


Fig. 3. An example of the sample placement process for 1 cm depths. (a) Thermocouple wires are placed ~16 mm below the designated depth, which is the depth of the sample placer tube; (b) sand is filled to the designated depth (1.54 g cm⁻³), and the sample placer is pressed into the sand; (c) sand is scooped out from the tubes, which are refilled with the PyOM-sand samples (~0.8 g cm⁻³); (d) the sample placer is lifted out with the PyOM-sand samples remaining; (e) a second set of thermocouple wires is placed on top of the PyOM-sand samples; and (f) the container is filled with sand to bury the samples (this step is omitted for surface treatments).

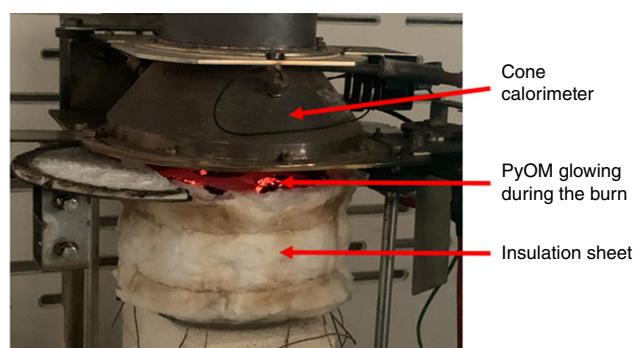


Fig. 4. Pyrocosm wrapped in white insulation sheet under the cone calorimeter (photo taken during the burn of High HF + Surface).



Fig. 5. Cooling the samples. Pyrocosm with sand-PyOM matrix is wrapped in water-circulating ring. Thermocouple wires can be seen attached to the pyrocosm.

cool. After the temperature of the samples dropped below 200°C, we slid a water-circulating ring onto the pyrocosm to speed up the cooling process and ensure the cooling was even for each replicate (Fig. 5). After the temperature dropped below 80°C, we took the pyrocosm out for sample collection. Each individual sample was collected and reserved for further chemical and biological analyses not described in this paper.

The cone calorimeter in our experiments has both temperature control (T control) and heat flux control (HF control) modes. The T control mode directly controls the temperature of the cone above the pyrocosm, while HF mode controls the emission of heat based on the heat flux that the sensor receives, which requires that the sensor be set at the same location as the samples. However, there was not enough space to place the heat flux sensor and the samples in the same pyrocosm. Additionally, since the controlling heat flux sensor is water-circulated for cooling, it could also cool the samples if they were placed together. Thus, we ran the calorimeter under T control mode during the burning of the samples. To ensure that we were able to administer the desired heat flux during the sample burns, we ran sand-only blank trials in HF control

mode for each heat flux treatment, where only the heat flux sensors were embedded in the pyrocosm (no samples), while we also recorded the temperature data (monitored by thermocouple in the cone calorimeter). Then we ran another blank trial under T control, using the temperature profiles recorded from HF control trials, and recorded the heat flux data (Supplementary Figs S4 and S5). Across three runs for each heat flux profile, heat flux tracked the setpoint closely (Supplementary Fig. S4), with deviations from setpoint generally $< 4 \text{ kW m}^{-2}$ (Supplementary Fig. S5). After ensuring that the heat flux data closely traced the prescribed heat flux profiles, we were able to run the calorimeter in T control mode during the experimental burns to achieve the same targeted heat flux. This blank-trial calibration process was repeated if the profiles had not been run for more than 3 months to ensure minimal instrumental drift and repeatability of the experiment.

Statistical analysis

Calculations and statistical analyses were done using Excel and R (R Core Team 2022). Figures were created using ggplot 2 in R (Wickham 2016). A two-way ANOVA with an interaction term for heat flux and burial depth and Tukey's HSD (Tukey 1949; Graves *et al.* 2019) were used to determine any significant differences between treatments. Degree hours were calculated from the area under the temperature profile over time. A binomial logistic model (function 'glm') (R Core Team 2022) was used to fit the degree hours and peak temperature as predictors for the fractional mass loss of PyOM.

Results

Qualitative observations

The PyOM samples in surface treatments under both heat flux profiles started to combust in the first 5 min. During the High HF + 1 cm treatment, combustion could not be observed directly because the samples were buried, but we visually observed smoke escaping from the pyrocosm within the first 17 min after the start of exposure. Thus, we expect that for those three treatments (High HF + Surface, High HF + 1 cm, and Low HF + Surface), the biggest PyOM losses might occur much sooner than the full heat flux profile's duration. (We were not able to directly track mass loss dynamically during the heating process due to oscillation caused by buoyancy-driven ambient airflow.) Those three treatments also resulted in little visible PyOM left (e.g. Supplementary Fig. S6). No obvious smoke was observed for High HF + 5 cm, low HF + 1 cm, or low HF + 5 cm.

Temperature profiles are consistent within and distinctive across treatments

Temperature profiles were consistent across the five PyOM samples within a given treatment and showed distinctive

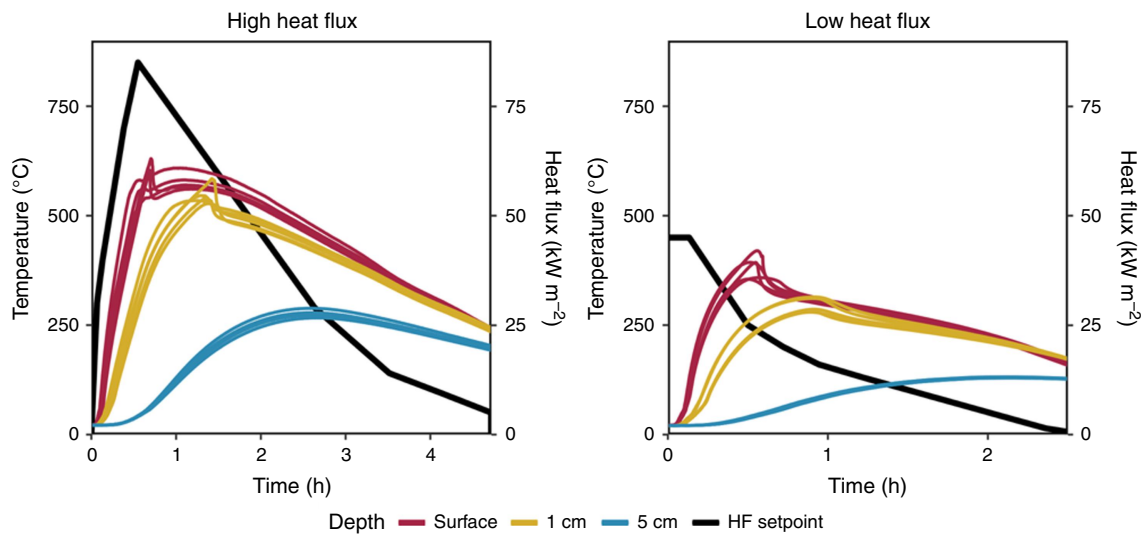


Fig. 6. Temperature profiles for the bottom thermocouples for five replicate samples in each HF treatment (coloured lines) and the corresponding heat flux profiles (black lines), for the High HF (left) and Low HF (right). The temperature data captured by the top thermocouples were not graphed due to excessive oscillations in the near-surface treatment as the thermocouples became exposed to the air (for full temperature profiles, refer to Supplementary Fig. S2). Note different time scales on x-axis. Samples continued to cool to room temperature beyond data plotted here.

Table 1. Mean degree hours and peak temperature data and statistical results ($n = 5$ PyOM samples per burn simulation; parentheses indicate standard error; Different superscript letters (ANOVA, Tukey’s HSD, $P < 0.05$) indicate significant differences across all treatments.).

Heat flux profiles		High			Low		
Heating duration (h)		4.72			2.5		
Exposure depth		Surface	1 cm	5 cm	Surface	1 cm	5 cm
Degree hours (°C × h)		2006.46 (57.67) ^A	1750.15 (49.32) ^B	928.27 (26.98) ^C	639.10 (10.90) ^D	539.47 (24.81) ^E	224.20 (3.04) ^F
Peak temperature (°C)		598.91 (23.64) ^A	546.39 (22.24) ^B	276.08 (8.03) ^D	383.85 (27.16) ^C	294.64 (16.66) ^D	130.24 (0.94) ^E

patterns for different HF treatments (Fig. 6). Overall, temperatures decreased with burial depth, and were higher in the High-HF treatments at the same depth (Table 1). The peak temperatures reached during the experiment had a time lag from the peak heat flux emitted from the cone calorimeter. The temperature profile and peak temperature of the High HF + 5 cm were similar to those of Low HF + 1 cm treatment (Fig. 6, peak temperatures of 276°C and 295°C, respectively). Peak temperatures were not significantly different between these two treatments but were significantly different among other treatments ($P < 0.001$, ANOVA in Supplementary Table S1, Tukey’s HSD, Table 1), and ranged within 130–599°C. The degree hours were significantly different across each treatment ($P < 0.001$, ANOVA in Supplementary Table S2, Tukey’s HSD, Table 1).

Higher heat fluxes and shallower burial depths lead to higher PyOM mass loss

Mass loss ranged from almost 100% (High HF + Surface and High HF + 1 cm, 98.86% and 98.45%, respectively) to as

little as 6.6% (Low HF + 5 cm) (Fig. 7). Mass losses for High HF + 5 cm, Low HF + Surface, and Low HF + 1 cm were 29.19%, 93.51%, and 47.72%, respectively (Fig. 7). (Note that the entire pyrocosm was weighed before and after the burn, so we report only the total mass loss of each treatment instead of individual samples.) Peak temperature during the burn was more strongly associated with mass loss than degree hours based on model fitness [indicated by pseudo- R^2 and Akaike information criterion (AIC) for model fitness; Fig. 8]. From the fitted curve, the bulk of mass loss (loss of 10–90%) occurred at temperatures within 185–415°C (Fig. 8).

Discussion

Simulating overhead soil heating in the laboratory

The High and Low HF profiles created distinctive differences in degree hours, peak temperatures, and PyOM mass loss. The pyrocosm setup resulted in consistent heat fluxes at a given depth, as indicated by the low variation among thermocouples

located at samples within a single run of the same treatment (Fig. 6) and low variations in heat flux across three 'blank' sand-only runs for each heat flux profile (Supplementary Figs S4 and S5). The results from the blank burns showed that the heat flux was typically within $\pm 4 \text{ kW m}^{-2}$ of the prescribed heat flux profile (Supplementary Figs S4 and S5).

Applications of the system and potential limitations

With this method of assessing the effects of fire on PyOM, we directly address the four limitations described in the

Introduction. (1) All portions of PyOM after the reburn are collected and analysed. The tradeoff is that the surrounding quartz sand matrix is also collected, which would affect some subsequent analyses, but not others, as described below. (2) PyOM is buried at a range of depths within a sand matrix, allowing the inclusion of often-overlooked mineral soil, while also ensuring that the PyOM sample is the only C input. (3) Instead of relying on a fuel bed to sustain heat during the burn, we apply a controlled and dynamic heat flux dose using a cone calorimeter, offering precise and reproducible conditions. This also ensures that no additional C inputs are introduced through fuel. (4) The heat flux profiles, representing high and low fire intensity, were based on real-world data captured from heat flux sensors beneath tree log burns, thus parameterising realistic fire scenarios. This method is general enough to be adapted to various fire-affected ecosystems and other forms of soil heating.

Matrix and burned samples

Using quartz sand as the matrix to contain the PyOM samples not only ensures chemical and physical uniformity, but also simulates the soil texture in typical jack pine barrens. Because quartz has a high melting point ($\sim 1700^\circ\text{C}$), is chemically inert even during extensive heating, and was ashed and washed before simulations, changes to the PyOM should be the only factor accounting for the difference in chemical properties for different treatments. This approach may be limited for finer soil textures and different mineralogy, where soil texture can change due to the fusion of clay minerals during heating (Badía and Martí 2003). Furthermore, such effects would complicate the heat transfer. Similarly, non-pyrogenic organic matter

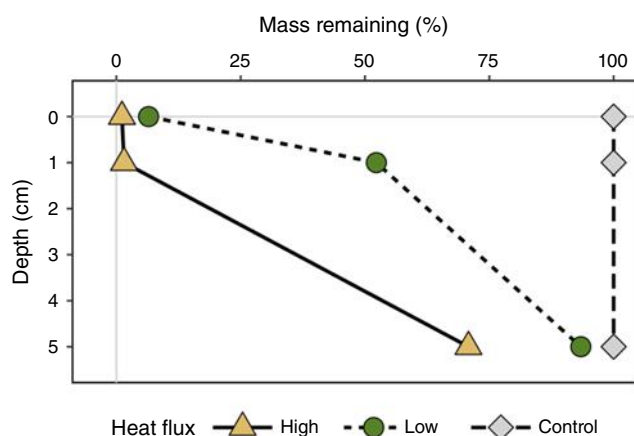


Fig. 7. Mean PyOM mass remaining (%) after each treatment ($N = 1$, representing mean of five samples within each heat flux $\times$ depth treatment). Note the y-axis is oriented to mirror the orientation of the sample (0 at the top, representing the surface of the sand matrix).

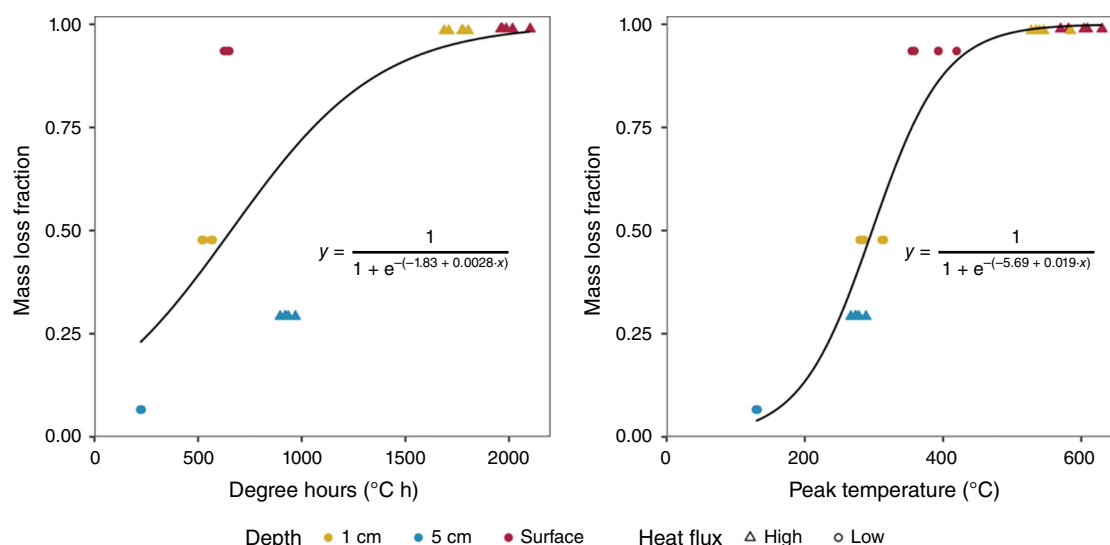


Fig. 8. Relationship between degree hours (left; curve indicates logistic model fit; $P < 0.05$ for all coefficients; pseudo- $R^2 = 0.05$; AIC = 31.316) or peak temperature (right; curve indicates logistic model fit; $P < 0.05$ for all coefficients; pseudo- $R^2 = 0.18$; AIC = 18.311) and mass loss fraction. $N = 1$ for mass loss fraction (fractional mass loss for all five samples in the pyrocosm) and $N = 5$ for degree hours and peak temperature (temperature was measured individually at the base of each sample).

naturally present in mineral soil, but not included in this simulation, could also be expected to affect heating dynamics in a natural soil. In systems where using quartz sand is appropriate, this method ensures a relatively consistent heat transfer due to uniform particle size, but further studies are needed to determine its suitability for different soil textures, mineralogies, and natural soils. This general setup could be readily applied to PyOM produced from different materials or at different temperatures, as well as unburned organic materials, to represent different ecosystems.

Moisture and oxygen content

Some moisture would be naturally present in most soils, even though the soil of jack pine barrens is typically sandy and well drained. However, moisture was not incorporated into the experiments for simplification purposes. In general, we predict that a moist sand matrix would have higher heat capacity due to the presence of water, so it would take more energy for the temperature to increase than the dry sand matrix. Massman (2012) modelled sand heating and moisture transportation and indicated that moisture (14% of the volume) at and above a depth of 35 mm in a sand matrix evaporated at 100°C within the first hour of heating, and the temperature surged after the water was evaporated. We might thus predict that the near-surface treatments in our experiments would not be dramatically different if the matrix were moist rather than dry as in this experiment; however, for samples at 5 cm, lower temperatures due to high moisture might decrease mass losses. An additional consideration is that water can also help create anoxic or low-oxygen (O₂) conditions that slow the oxidation of PyOM during heating. Although the O₂ content in the pyrocosm was not directly measured, the general burn conditions for surface treatments were assumed to be aerobic. In theory, when surface heating is sufficient to initiate combustion >200°C, gas products tend to move upwards and exit the pyrocosm, and the replenishment of O₂ at deeper soil profiles is much slower than its depletion (Bryant *et al.* 2005). Thus, we should likely assume that the samples in the buried treatments have experienced both oxic and anoxic heating.

Small-scale application and parameterising heat flux

Even within a single fire, burning effects can be highly spatially heterogeneous (Brucker *et al.* 2022). This method is specifically designed to simulate a single heat flux profile, and thus would not be appropriate on its own within a single run for simulating spatially heterogeneous effects of heating. However, the results of different heat fluxes in different runs could potentially be combined using a modelling approach.

To measure heat fluxes as we parameterised our burns, we used water-circulated heat flux sensors, which would be difficult to deploy in the field; however, simple ones, such as directional flame thermometers (Najafi *et al.* 2015), could be relatively easily deployed during a prescribed fire. The cone calorimeter we used can also dose constant heat fluxes

if desired instead of dynamic profiles. For example, a short constant heat flux could be used to simulate the soil surface heat flux of a fast-moving crown fire (Johnson *et al.* 2023).

Possible analyses

This approach could be used to simulate realistic soil heating from above in a depth-resolved way (as contrasted with putting a soil sample into a muffle furnace, for example). While this methods-focused study considers only temperature profiles and mass loss, the resulting samples could be analysed using a range of methods. Because the sand contains negligible organic matter, the combined sand–PyOM samples can be analysed for total elemental composition, such as for C and N, and the total C and N losses due to the burns can be readily calculated. Other chemical properties, such as those inferred from spectroscopic analysis, would be complicated by the presence of sand mixed with the PyOM, and it may be difficult to ensure a sufficiently strong signal derived from the PyOM (especially with high levels of combustion) while also accounting for a pure sand ‘blank.’ In such cases, the scientist could choose to not mix PyOM with sand in the sample holder and/or try to collect the most PyOM-rich portion of the sample, without attempting to capture all PyOM in the sample. For non-quantitative chemical characterisation, this might work, especially if unburned control samples are included as a comparison. Chemical properties such as pH, electrical conductivity, or dissolved organic C could be assessed on the combined sand–PyOM samples, but will yield different values than if conducted on pure PyOM without sand. In such cases, the trends across samples should be interpretable, but the absolute values may or may not be relevant (depending on how the sand blank affects measurements). Finally, for biological analyses, biological availability of resulting PyOM-C could be determined by incubating the PyOM–sand sample with microbial communities extracted from fire-affected soils. Again, with this approach, absolute values may not be so directly applicable, while trends across samples are more likely to be relevant. Alternatively, the burned PyOM could be extracted from the sand matrix (although would likely retain some sand) and added directly to soils for incubation. This would work particularly well if the PyOM were ¹³C-labelled, to allow it to be conclusively distinguished from soil C.

Even buried PyOM can still be combusted in subsequent fires

Consistent with our hypotheses, higher heat fluxes and shallower depths had greater PyOM mass losses. Previous laboratory and field experiments by Doerr *et al.* (2018) (boreal forest wildfires) and Bartoli *et al.* (2021) (laboratory burns under a bed of litter) determined that low-intensity fires consumed 17–50% of PyOM in mass at the litter surface and soil surface, while high-intensity fires consumed 50–84%. In our study, the mass loss of PyOM at the surface was substantially

higher – 93.51% under the Low HF profile (equivalent to low-intensity fire) and 98.86% under the High HF profile (equivalent to high-intensity fire). Our buried PyOM had substantial mass loss (>25%) for all buried treatments, except for 5 cm depth under Low HF profile. This likely reflects the fact that our study was designed to represent the upper end of heat fluxes characteristic of realistic surface burns. Therefore, our results would not be a good estimate for total PyOM mass loss across a typical burn on a landscape scale but, rather, represent local conditions where logs are burning on the forest floor.

In wild-land fires, fire may move quickly on a landscape due to various factors such as wind and topography, so the heating duration could be shorter than the heat flux profiles we applied here. At spots where the fuel density is high, the fire may dwell longer. However, heating duration may not be a key determinant of PyOM mass loss after a certain amount of time, as we found that ‘degree hours’ was a poorer predictor of mass loss than peak temperature (Fig. 8). Another limitation to our approach for estimating PyOM losses is that ageing of PyOM between fires would occur over time (Wozniak *et al.* 2024), whereas here we simulated the subsequent burn shortly after producing the PyOM. It would be interesting to consider the intersections between PyOM ageing and the timescales and effects of reburns in a future study, either by simulating PyOM ageing (Zeba *et al.* 2022) or through field studies.

Tinkham *et al.* (2016) indicated that PyOM is easily degraded by subsequent fires when on the surface or shallow-buried, while thermal degradation is less likely in mineral layers (>30 mm) due to soil insulation. The exposure depths in our study were designed to interrogate this concept. However, substantial thermal degradation still occurred at the depth of 5 cm, particularly under High HF. Low-temperature heating (<300°C) can also increase C degradability by microbes (Norwood *et al.* 2013; Santín *et al.* 2016a). Most carbonaceous material starts to be combusted at 150–250°C (Baldock and Smernik 2002; Badía and Martí 2003), consistent with the temperature range where we began to see mass losses in our experiment (Fig. 8). Due to its heterogeneous nature, it is not possible to determine an exact temperature required for PyOM to be thermally degraded. Based on our results, even PyOM at 5 cm under Low HF, which had a peak temperature of <150°C (Table 1), experienced some mass loss. For all other treatments, the temperature stayed above 150°C for an extensive time (Fig. 6), which resulted in substantial thermal oxidation. Based on the logistic model fitting, we found that the bulk of mass loss of PyOM occurred in treatments with peak temperatures of 185–415°C, and stayed basically unchanged for the higher temperatures (due to near-complete combustion). Consequently, we could predict that PyOM even at 5 cm depth in soil profiles will be susceptible to consumption in subsequent fires, as long as the temperature reaches the limits required for thermal degradation. Supporting this, a recent study also found one-third of C losses occurred in the

mineral soil, with significant losses occurring even in the 15–30 cm depth after a high-severity wildfire (McCool *et al.* 2023).

Conclusions

We presented detailed methods for simulating soil heating from above, as applied to the effects of reburns on PyOM stocks. Using heat flux parameterisations with log burns and applying the heat flux profiles to PyOM using a cone calorimeter, the methods we designed for heat flux simulation should be replicable and provide distinctive temperature profiles for each treatment, and could be adopted for other soil heating experiments. We found that deeper burial depths can help protect PyOM from loss in the subsequent fires, and burial is a more effective protector under low-intensity fire. In future work, chemical and biological properties of the PyOM collected from the pyrocosm will be assessed using different laboratory tests to further analyse the net effects of fire.

Supplementary material

Supplementary material is available [online](#).

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Data availability. Data presented in the paper can be accessed online through the US Department of Energy’s ESS-DIVE repository at doi: [10.15485/2514357](https://doi.org/10.15485/2514357). Code used for data analysis and creating figures is available at <https://github.com/MengmengLuo/Reburning-pyrogenic-organic-matter-A-laboratory-method-for-dosing-dynamic-heat-fluxes-from-above>.

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