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SHORT COMMUNICATION

Influence of fire retardant and pyrogenic carbon on microscale changes in soil nitrogen and phosphorus

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Abstract During wildfire season in the western US, fire retardant chemicals are dropped from aircraft in an effort to control the spread of fire. Fire retardant dropped on sites that are not actively burning results in exceptionally high soil nitrogen (N) and phosphorus (P) "fertilization" effect on wildland soils impacting terrestrial and aquatic ecosystems. Herein, we used microdialysis to evaluate the short-term spatiotemporal dynamics of soil inorganic N and ortho-P fluxes in response to wood pyrogenic carbon (PyC) on soils receiving fire retardant (Phos-Chek) in a 28-d column experiment. Retardant additions to soil induced dramatic increases in soil inorganic N and ortho-P flux rates. The addition of wood PyC to soils with retardant significantly and immediately reduced ortho-P flux rates at multiple depths (1, 5, and 10 cm) and reduced NH_4^+ flux rates at 10 cm while retaining flux rates at 1 and 5 cm. These effects were observed throughout the course of the experiment and were more pronounced towards the end of the experiment. By d-28, PyC significantly reduced the NH_4^+, NO_3^-, and ortho-P accumulation on ionic resins at soil column bottoms by 50%, 52%, and 43%, respectively. The application of wood PyC may be effective at buffering excess nutrient fluxes from fire retardant and reduce short-term nutrient leaching in unburnt forest soils exposed to fire retardant.

Keywords Charcoal · Firefighting · Fire retardant · Microdialysis · Nutrient leaching · Pyrogenic carbon

Introduction

Nitrogen (N) and phosphorus (P) rich fire retardants are commonly used to suppress wildfires in the western US, yet there is currently limited knowledge of how fire retardant used in fighting wildfires influence soil nutrient dynamics. Fire retardants are chemical products consisting of flame inhibiting chemicals that, either applied alone or mixed with water, delays or stops the combustion of a given fuel (Bourbigot and Duquesne 2007). These substances are generally composed of fertilizer salts with a thickening agent or a mixture of surfactants, wetting agents and solvents. A commonly used retardant chemical, Phos-Chek D75R for example, contains 65%

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ammonium sulfate, 15% ammonium phosphate, and 5% diammonium hydrogen phosphate (on a dry weight basis) (Gould et al. 2000). When delivered at an operational coverage of approximately 1 L m^{-2} to fight a fire of 2000 kW m^{-1} intensity, it has been estimated that the nutrient inputs from the retardant drop will result in a soil application rate of 337 kg ha^{-1} N and 94 kg ha^{-1} P (Bell et al. 2005). In the United States, approximately 77 million liters of fire retardant is dropped every year on soils that normally would not receive fertilizers (Aviation Aircraft Use Summary, US Forest Service). In California, state firefighting crews have applied over 50 million liters of fire retardant in 2017 alone. Such large nutrient pulses along with the fire retardant do not naturally occur in wildland forest settings in the western US and can result in significant impacts on both aquatic and terrestrial ecosystems including acute toxicity to fish in lakes and streams (Kalabokidis 2000) or a net decline in vegetation species richness in riparian areas (Larson et al. 2000). The legacy effect of this N and P enrichment has also been found to last as long as nine years following a single retardant drop (Marshall et al. 2016). Significant soil N and P leaching events following fire retardant drops were also reported to last for more than a month (Pappa et al. 2006, 2007). To date, few studies have taken any steps to explore potential mitigation practices to be employed following fire retardant derived “fertilization” effects (Santín et al. 2020).

Biochar is pyrogenic carbon (PyC) specifically intended as a soil amendment and has been widely demonstrated to absorb and adsorb nutrients thereby reducing net leaching rates in various ecosystems (Gao and DeLuca 2016, 2020). In this study, we evaluated the influence of PyC on soil N and P fluxes following applications of a N and P rich fire retardant to surface mineral soils. The PyC used in this study served both as a proxy for naturally formed PyC generated during a wildfire and for biochar that might be added to soils after retardant drops to reduce nutrient losses (Bird 2015; Woolet and Whitman 2020). We hypothesized that adding PyC to forest soils that received fire retardant would reduce nutrient leaching by adsorbing N and P thereby reducing nutrient flux in surface soils. Microdialysis is an emerging technique being utilized in soil science to allow for in situ evaluation of solutes with minimal disturbance to the soil environment, thus is able to provide high spatiotemporal resolution of a target solute’s flux dynamics, for example inorganic N or ortho-P (Shaw et al. 2014; Demand et al. 2017; Buckley et al. 2017, 2020; Hill et al. 2019; Gao and DeLuca 2019). Herein, microdialysis was used in a soil column study to monitor fine scale diffusive flux rates of NH_4^+, NO_3^-, and ortho-P along a soil column following PyC addition to a typical western US forest soil with or without fire retardant drop over a 28 day period.

Materials and methods

Sampling site and soil description

Soils were collected from the A and upper Bw horizons (lower depth 5 cm and 25 cm, respectively) of an undisturbed site at the Lubrecht Experimental Forest, Greenough, Montana, USA (46°53′N, 113°23′W). The sampling site is located in the northwestern Rocky Mountain Range and experiences a temperate semi-arid climate (mean annual precipitation 480 mm; mean annual temperature 5.5 °C). Dominant vegetation of the region includes Douglas-fir (*Pseudotsuga menziesii*), ponderosa pine (*Pinus ponderosa*), and lodgepole pine (*Pinus contorta*). Soils (Inceptisols, Typic haplocryepts, sandy loam texture) originated from colluvium and are well-drained (NRCS Soil Survey 2020) and slightly acidic (pH of 6.5 ± 0.2). This forest region and its soils are considered representative of western US fire-prone zones and thus may have the potential of receiving fire retardant during fire seasons (Gundale et al. 2005; Fiedler et al. 2010).

Experimental design

Soils collected from two horizons (A and Bw) were thoroughly homogenized and mixed, and pre-moistened to approximately 60% field capacity, passed through a 2-mm sieve, packed into 4 cm diameter by 20 cm length PVC column containers with gentle compaction that resulted in an evenly distributed soil profile. The resulting “control” soil column (no PyC or fire retardant) had a bulk density of 1.1 g cm^{-3}. Treatment columns were prepared by applying: (1) Fire retardant (FR): 1.5-ml fire retardant uniformly at surface soils; (2) Pyrogenic carbon (PyC): 5.8 g PyC (equivalent to 2% w/w) at surface soils (mixed into top 0–3 cm with gentle compaction); and (3) Fire retardant followed by PyC (FR + PyC): PyC (2% w/w) on soils received 1.5-ml fire retardant (PyC was applied 4 h following retardant drop and was gently mixed with retardant and soils at 0–3 cm soil depth). Each control or treatment was replicated four times (n = 4) resulting in a total of 16 columns. Control and PyC columns all received 1.5-ml DI water. The fire retardant suspension was thoroughly shaken before and during column applications to ensure consistency. The amount of retardant added to each column was calculated based on the surface area of the column and the application rate commonly used on field site with light fuel loads (e.g., 12.2 m^3/ha for low severity surface fire or grassland fire, equivalent to approximate 162 kg N ha^{-1} and 326 kg P ha^{-1}, see Marshall et al. (2016) for more information).

PyC and fire retardant description

PyC was produced using charred wood waste from lumber mills of F.H. Stoltze Land & Lumber Co. (Columbia Falls, Montana, USA) as a byproduct from the electrical co-generation plant (https://www.fhstoltze.com/; https://genesisbiochar.com/), and was further press processed and sieved to 5-mm before application. The feedstock of wood PyC was a mixture of Douglas-fir (*P. menziesii* L.), western larch (*Larix occidentalis* L.), grand fir (*Abies grandis* L.), subalpine fir (*Abies lasiocarpa* L.), and lodgepole pine (*P. contorta* L.); PyC generation temperatures were observed to be in the range of 450–550 °C (personal communication). The fire retardant used in this study was LTFR Phos-Chek P100-F (ICL Performance Products, CA, USA), a product commonly used by US Forest Service during wildfire seasons (www.fs.fed.us/rm/fire/wfcs/products/index.htm). The fire retardant comes in a mixed liquid form that is prepared from a dry concentrate formulation which contains approximately 76–82% (w/w) $NH_4H_2PO_4$ and 8–12% (w/w) $(NH_4)_2HPO_4$ (www.phoschek.com). Physicochemical properties of soil, PyC, and fire retardant used in this study are listed in Table 1.

Microdialysis sampling and chemical analyses

Each column had three small holes (by 1 mm diameter drill bit) bored at depths of 1, 5, and 10 cm to allow access by the microdialysis probe. Each treatment or control column represented an individual sampling unit where three measurements were taken from each column at a given depth on the same sampling day and were averaged to generate column-level values accounting for within-column variation. One ionic resin capsule (mixed anion and cation resin, UNIBEST Ag Manager, WA, USA) was installed at the bottom of each column container (20 cm depth) during packing to track the cumulative N or P leaching. All columns were placed in a greenhouse and maintained at 22 °C (± 6 °C) over the course of the experiment. We added 64 ml DI water to all soil columns (resulting in ~ 22–25% saturation) at a single time one day following treatment applications (day 2) to simulate the monthly average precipitation (May). Column surfaces were not covered and water loss by evaporation was not further adjusted during the experiment in an attempt to simulate field conditions and eliminate the impact of drying-rewetting or leaching with water addition. Microdialysis probes used in our study had 10 mm membrane length, 500 μm membrane diameter with a 20 kDa molecular weight cutoff (CMA 20, Harvard Apparatus). We used the exact same procedures for microdialysis calibration and dialysate sampling and collection as described in a previous study (see Gao and DeLuca 2019). A total of nine samples were collected for an individual column (3 samples per depth × 3 depths). All dialysates were first sampled 96 h following treatment addition (day 4), and were then sampled every four days for a total of 28 days (day 8, 12, 16, 20, 24, and 28). Dialysate NH_4^+ and NO_3^-, and ortho-P were determined colorimetrically following Mulvaney et al. (1996), Miranda et al. (2001), and Ohno and Zibilske (1991) using a microplate spectrophotometer (BioTek Instruments Inc., VT, USA). The resulting concentration data (mg L^{-1}) were converted to diffusive flux rate expressed as nmol N or P cm^{-2} h^{-1} following Inselsbacher et al. (2011). Resin capsules were retrieved at day 28 and extracted following Gao et al. (2016) and MacKenzie and DeLuca (2006) before N and P analyses using methods described above.

Statistical analyses

Microdialysis data on multiple sampling days and at multiple soil depths were analyzed and visually

Table 1 Characteristics of pyrogenic carbon, fire retardant, and soil used in this study

	pH	NH_4^+-N	NO_3^--N	Ortho-P	Total C	Total N	Total P
Pyrogenic carbon	8.0	0.14 mg kg^{-1}	N/A[a]	0.66 mg kg^{-1}[b]	595 g kg^{-1}	0.87 g kg^{-1}	2.42 g kg^{-1}
Soil	6.5	5.56 mg kg^{-1}	0.68 mg kg^{-1}	9.09 mg kg^{-1}[b]	16.5 g kg^{-1}	1.4 g kg^{-1}	0.98 g kg^{-1}
Fire retardant	5.0	17,500 mg L^{-1}	145 mg L^{-1}	32,500 mg L^{-1}	2.91 g L^{-1}	18.8 g L^{-1}	33.4 g L^{-1}

[a] Below instrument detection limit

[b] The ortho-P in pyrogenic carbon or soil was determined colorimetrically using the malachite-green method (Ohno and Zibilske 1991) following 0.01 *M* $CaCl_2$ extraction targeting soluble diffusive inorganic P (DeLuca et al. 2015a)

presented separately to demonstrate the spatiotemporal dynamics. Microdialysis flux rate data were first analyzed using 3-way repeated measures ANOVA to reveal the effect of treatment, sampling day, sampling depth, and their interactions at the significant level of $P = 0.05$. To reveal how soil nutrient fluxes are affected by treatments at a given soil depth over time, microdialysis flux rate data at a given depth were compared across treatments and sampling days using a 2-way repeated measures ANOVA with significance accepted at $P \leq 0.05$. Tukey's post-hoc test was subsequently conducted to give pair-wise comparisons. The same method was applied to resin data comparing across treatments at $P = 0.05$ (n = 4). All data were evaluated for normality and homoscedasticity prior to analysis and all data sets met the assumptions of parametric statistical methods. Data analyses were conducted using R (R Core Team 2016).

Results

Soil nutrient fluxes were greatly influenced by the application of fire retardant and fire retardant with PyC. A significant interactive effect between treatment and sampling day was observed at nearly all sampling depths for NH_4^+ and ortho-P flux rates; whereas that interactive effect was non-significant for NO_3^- flux rates regardless of sampling depth (Figs. 1, 2, 3, Online Resource 1 Table S1). Specifically, significantly higher NH_4^+ and ortho-P flux rates (about 4–5 times higher than control) were observed at all depths following retardant addition on unburnt soils throughout the 28-d experiment (Figs. 1, 2), supporting our hypothesis that the fire retardant delivers an "extreme fertilization" effect (*P* (treatment) < 0.001 at all depths for both NH_4^+ and ortho-P). Dialysate NO_3^- flux rates were not significantly influenced by the fire retardant or PyC spatiotemporally over the course of the experiment (Online Resource 1, Figure S1, *P* (treatment × sampling day) $\geq$ 0.1 at all depths).

PyC used on soils without retardant had a limited effect on either N or P flux rates regardless of sampling day or depth (Figs. 1, 2, Online Resource 1 Figure S1). By contrast, NH_4^+ flux rates at 10 cm depth in "FR + PyC" were significantly lower than that of "FR" across all sampling days, and this PyC effect was more pronounced towards the end of the experiment (Fig. 1c, *P* (treatment x sampling day) < 0.01). However, the flux rates of NH_4^+ at surface (1 and 5 cm) in soils with fire retardant generally exhibited minimal response to PyC additions throughout 28-day (Fig. 1a, b). Similarly, ortho-P flux rates in "FR + PyC" soil were significantly lower than "FR" at all soil depths throughout the sampling period despite being higher than the control (Fig. 2, *P* (treatment) < 0.001 and *P* (sampling day) > 0.10 at all depths).

Fire retardant application to soil resulted in greater N and P downward translocation, but the addition of PyC significantly reduced the amount of NH_4^+, NO_3^- and ortho-P accumulated on ionic resins at the bottom of the soil columns at d-28 by 50%, 52%, and 43%, respectively (Fig. 3). It is also important to note that adding PyC to soils receiving fire retardant retained surface soil (1–5 cm) pH at its background level (pH 6.6 ± 0.3) by the end of the 28-day incubation period (Online Resource 1, Table S2).

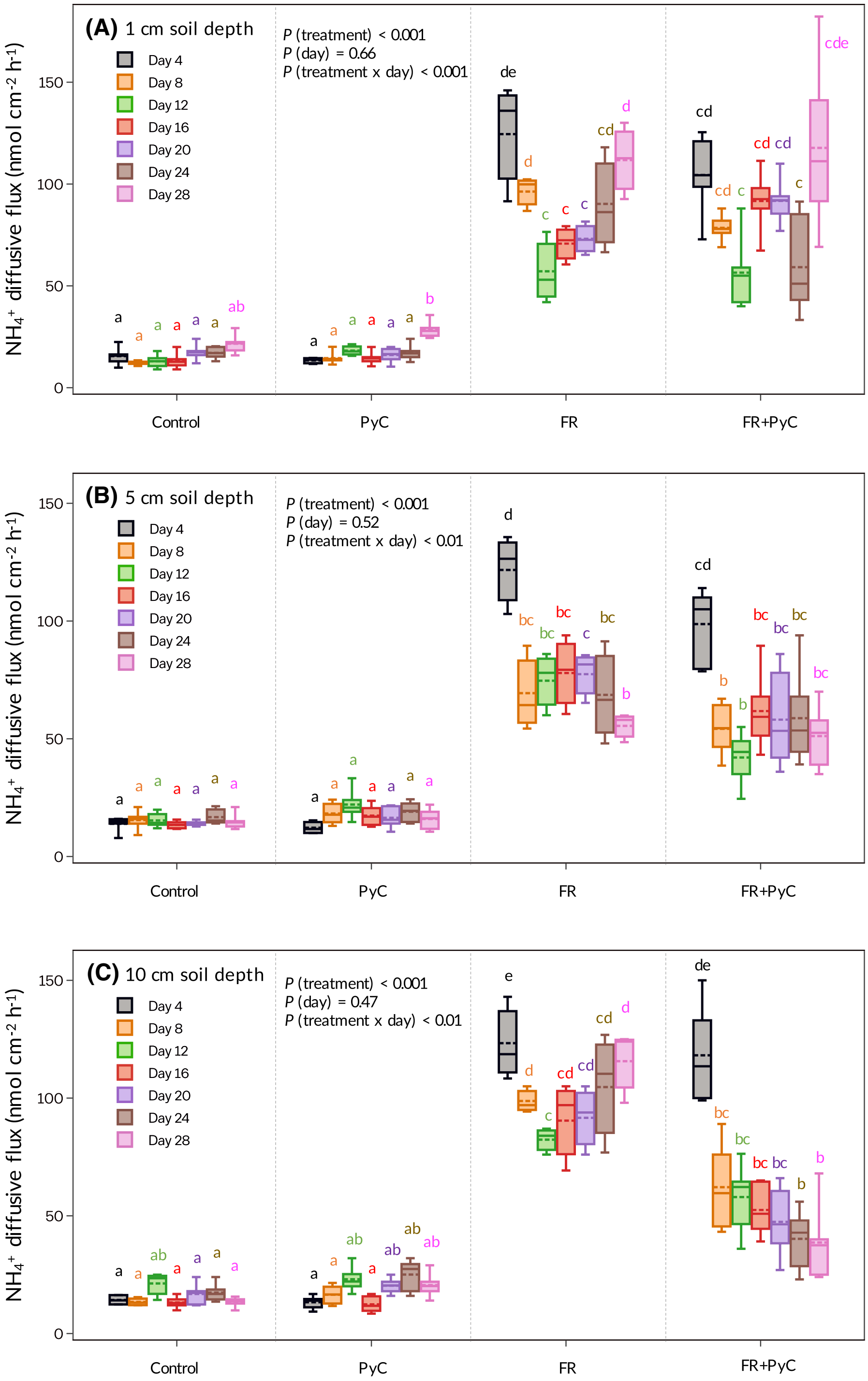


Fig. 1 Soil ammonium (NH_4^+) diffusive flux rates (nmol N cm^{-2} h^{-1}) sampled by microdialysis at **a** 1 cm, **b** 5 cm, and **c** 10 cm depth in a soil column amended with no pyrogenic carbon or retardant (control), pyrogenic carbon (PyC), fire retardant (FR), and fire retardant and pyrogenic carbon (FR + PyC) in an incubation experiment (n = 4). Sampling was conducted at Day 4, 8, 12, 16, 20, 24, and 28 following treatment application. Data at individual depth were compared using Tukey-HSD test following ANOVA. Boxes with the same letter are not significantly different at $P = 0.05$. Solid line indicates median, dashed line indicates mean

Discussion

Fire retardant application to surface soils significantly increased inorganic N and ortho-P fluxes during a 28-day incubation experiment; however, PyC application to these same soils eliminated the effect of fire retardant on nutrient fluxes. This finding indicates that PyC has the potential to reduce downward

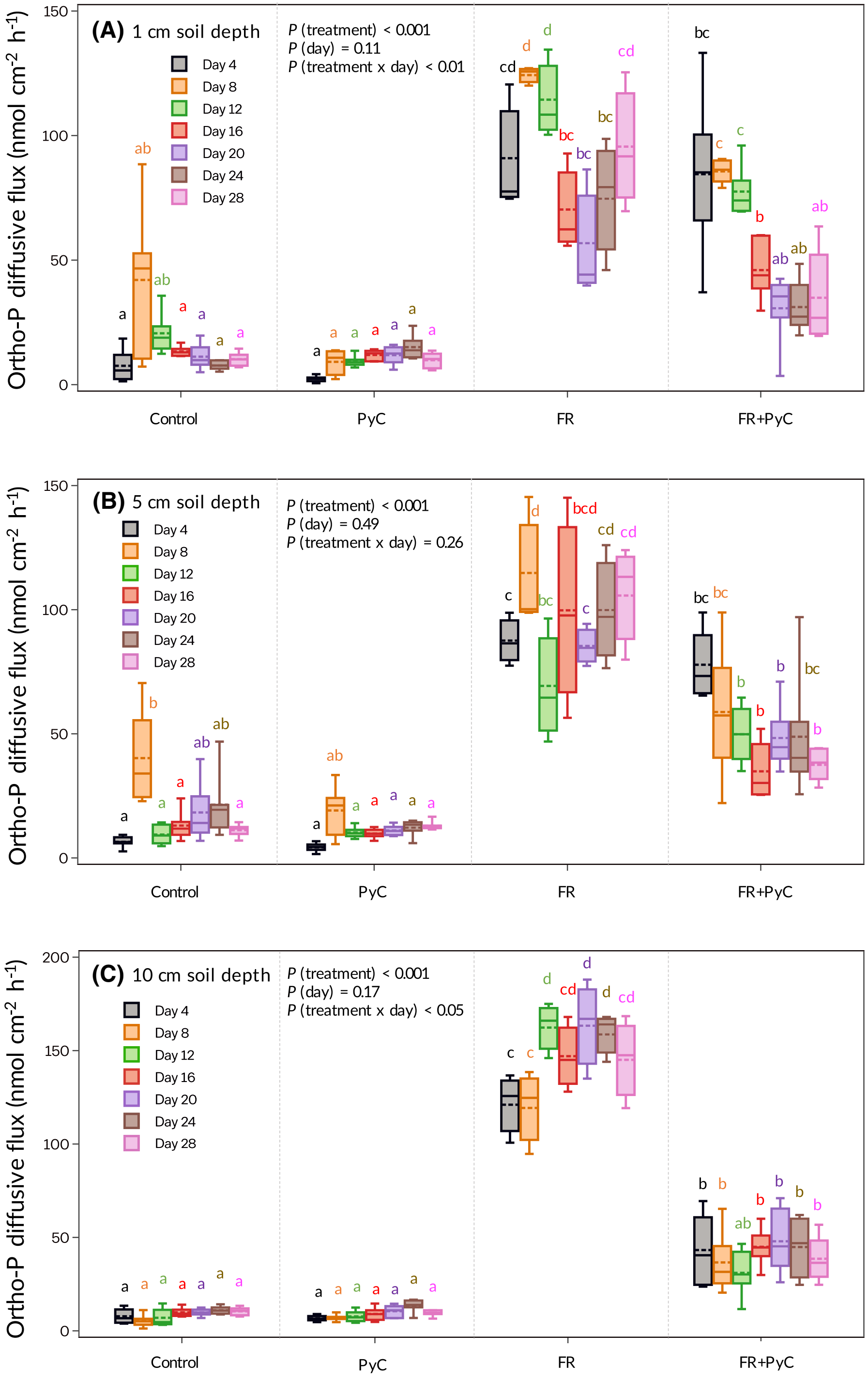
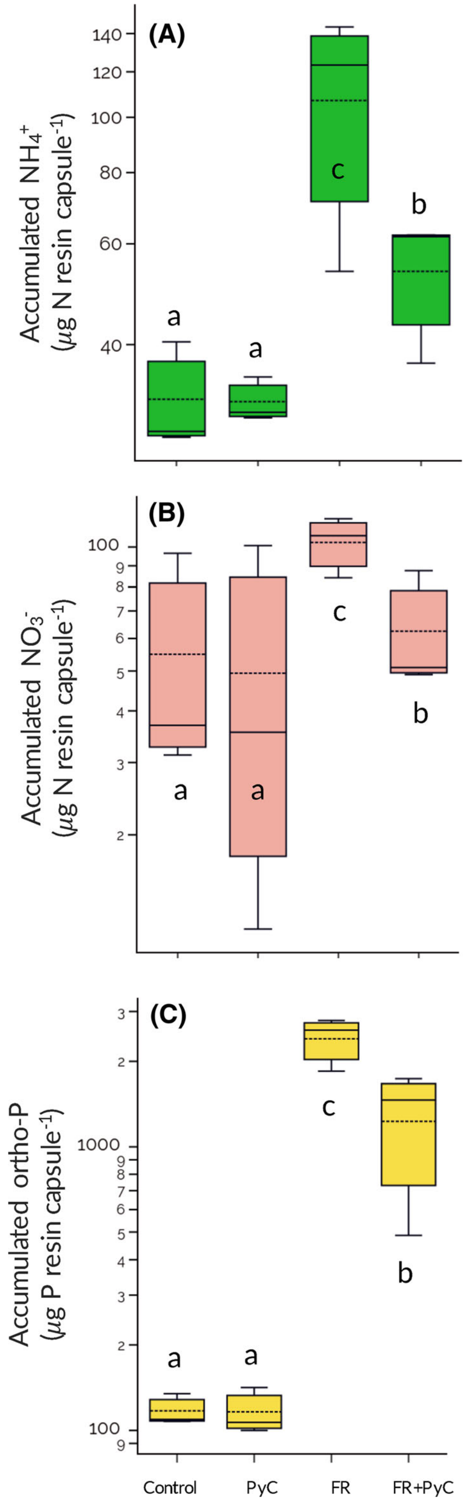


Fig. 2 Soil ortho-P diffusive flux rates (nmol P cm^{-2} h^{-1}) sampled by microdialysis at **a** 1 cm, **b** 5 cm, and **c** 10 cm depth in a soil column amended with no pyrogenic carbon or retardant (control), pyrogenic carbon (PyC), fire retardant (FR), and fire retardant and pyrogenic carbon (FR + PyC) in an incubation experiment (n = 4). Sampling was conducted at Day 4, 8, 12, 16, 20, 24, and 28 following treatment application. Data at individual depths were compared using Tukey-HSD test following ANOVA. Boxes with the same letter are not significantly different at $P = 0.05$. Solid line indicates median, dashed line indicates mean

translocations of N and P from fire retardants added to surface soils.

The mixing of soil, fire retardant and PyC at the beginning of the experiment may have enhanced NH_4^+ adsorption to the PyC surface thereby excessively reducing the downward translocation of NH_4^+. This finding is consistent with numerous previous observations where PyC was found to interact with



◀**Fig. 3** Total **a** NH_4^+ **b** NO_3^- and **c** ortho-P accumulated at Day 28 on ionic resin capsules placed at the bottom of soil column for the control, pyrogenic carbon (PyC), fire retardant (FR), and fire retardant and pyrogenic carbon (FR + PyC) treatments in an incubation experiment (n = 4). Note log scale. Treatments were compared using Tukey-HSD following ANOVA. Boxes with the same letter are not significantly different at $P = 0.05$. Solid line indicates median, dashed line indicates mean

inorganic N fertilizer and enhance N retention in surface soils (Gao et al. 2017; Joseph et al. 2018). The finding that NH_4^+ flux was significantly reduced by PyC at 10 cm, but not 1 cm or 5 cm, and that this response was more pronounced toward d-28 highlights the spatiotemporal effect of PyC at altering NH_4^+ fluxes in soils with fire retardant. It is possible that NH_4^+ adsorbed onto PyC surface is actively engaged in an sorption/desorption equilibrium resulting in limited change in NH_4^+ fluxes at 1–5 cm depth within 28-day (Wang et al. 2015a). It is also important to note that the manipulation of soils in the experimental setup (e.g., sieving, mixing, and packing) could have initially promoted ammonification and nitrification, but these effects are clearly overwhelmed by the NH_4^+ additions associated with the retardant.

The lack of treatment effect on dialysate NO_3^- flux rates over the course of the experiment is possibly associated with the lack of NO_3^- input associated with fire retardant or the added PyC (Table 1), and the lack of net nitrification observed in microsites over time and space by the microdialysis system (Gao and DeLuca 2019; Buckley et al. 2020). Given that NO_3^- is highly mobile in soil solution, it is expected that NO_3^- flux or net nitrification occurred at individual depth is insensitive to time or treatment. However, the reduction in NO_3^- accumulation on resin capsules placed at the bottom of the columns with PyC suggests that PyC still had the potential to reduce the losses of NO_3^-, potentially due to NH_4^+ adsorption to PyC temporarily reducing or eliminating NH_4^+ as a substrate for nitrification in the soil solution (Wang et al. 2015b).

PyC likely sorbed ortho-P introduced by the fire retardant and precipitation of inorganic P with alkaline earth metals, e.g., Ca or Mg as provided by PyC (possibly due to the ash component, PyC pH is 8, Table 1) may have also induced ortho-P removal from soil solution (Cheng et al. 2006; DeLuca et al. 2015b). It is important to note that the fire retardant is mildly acidic (pH 5) and the oxidation of NH_4^+ added with fire retardant would further drop soil pH. The slightly acidic soil (pH 6.5) used in this study became more acidic (pH 6.0 ± 0.1) with retardant, but the addition of PyC to soils with fire retardant shifted surface soil pH back to pH 6.6 ± 0.3 after 28 days. It is possible that this soil pH change has shifted the distribution of ortho-P forms in soil solution (from predominantly $H_2PO_4^-$ to a mix of HPO_4^{2-} and $H_2PO_4^-$) (Price 2006) which may or may not promote the PyC-sorption and P removal process (Gao et al. 2019). Wood PyC may also contribute to the P removal process following a soil pH change (Xu et al. 2013) in spite of having an ortho-P content of 0.66 mg P kg^{-1}. Alternatively, PyC additions may have increased bulk soil C:P ratios compared to columns with fire retardant, thus some of the solution ortho-P might have been biologically removed by microbes through microbial P immobilization over 28-day (Zhang et al. 2018). The forest soil used in our study is relatively immature, where the coarse textured soil is not considered to be P-poor (Table 1). Therefore, it is unlikely that the sorption of Fe- and Al-oxide and hydrous oxides would limit P availability in this system and would increase the potential for P loss. Our findings highlight the effectiveness of PyC at reducing these excess P fluxes and losses within at least days to weeks.

To our knowledge, this is the first study to address the interactive effects of PyC and fire retardant chemicals on soil nutrient dynamics. Previous short-term studies conducted on soils using standard soil extraction methods often demonstrated an increase in surface soil nutrient retention and a decrease in leaching losses with PyC amendments (Nguyen et al. 2017; Gao et al. 2019). Our results describe a similar pattern with fine-scale spatiotemporal resolution. Microdialysis allowed us to detect and highlight the temporal changes in inorganic N and ortho-P fluxes at a fine scale (1, 5, and 10 cm depth) and advance a better understanding of nutrient flux mechanisms not attainable with bulk soil extraction studies. However, size, scale, and length of the study was somewhat constrained by the sampling capacity of the microdialysis system (i.e., a set number of sampling probes equipped with a single sampling unit, and relatively long sampling time with slow infusion of perfusate to ensure the effectiveness of probe calibration and sampling accuracy).

Overall, our study suggests that unburnt forest soils exposed to fire retardant may experience lower net NH_4^+ and ortho-P flux rates if soils are amended with PyC (wood biochar) after being exposed to fire retardant. This short-term (28-d) soil column experiment may help us understand how PyC might be applied in post fire recovery efforts to modify soil nutrient pulses induced by fire retardant applications. Finally, it is important to note that the findings in this study may not directly apply to naturally formed PyC given the variable conditions (temperature, moisture, source material) under which PyC is formed in the natural environment (Pingree and DeLuca 2017). A larger-scale field study involving either commercial PyC or wildfire-generated PyC would help advance additional insights for addressing the impacts of fire retardant use on forest ecosystem function.

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