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# Biochar selection for removal of perfluoroalkyl substances from reclaimed water for agricultural irrigation

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## Abstract

In agricultural contexts, the presence of per- and polyfluoroalkyl substances (PFASs) in recycled water may pose a threat to irrigated crops and the human food chain. Biochar potentially represents a low-cost and environmentally friendly approach to removing PFAS from recycled water via adsorption. Identifying key biochar properties responsible for successful adsorption of long- and short-chained PFAS as well as developing predictive models are crucial in identifying the potential of biochar as a scalable approach for remediating PFAS in agriculture. Here, a systematic and comprehensive evaluation of 17 physicochemical properties of 24 biochars was performed to determine the main factors influencing PFAS removal from water. Batch studies were conducted, and principal component analysis as well as correlation studies were used to determine factors influencing PFAS removal. Various parameters were influential in the removal of long- (carbon/nitrogen ratio, specific surface area) and short- ([nitrogen + oxygen]/carbon ratio, carbon/nitrogen ratio) chained PFAS. Using these 24 biochars as a training dataset, linear models were constructed to predict the removal of selected PFAS based on biochar properties. These models were used to select a commercial pine wood biochar (Rogue biochar), which performed effectively in removing PFOS, PFOA, PFBS, PFHxS, PFNA in different matrices. Post-pyrolysis thermal treatment facilitated maintenance of adsorption potential over subsequent cycles, while providing the additional benefit of increasing the removal of the short-chained sulfonate PFBS by two- to five times. Careful, evidence-based selection of biochars with optimal physicochemical characteristics can provide excellent removal of both long- and short-chained PFAS compounds from water.

## Highlights

- Removal efficacy of PFAS from water was related to certain physicochemical properties of biochar (SSA, C, N, and O).
- Linear models may be suitable for predicting PFAS removal performance of biochars based on their properties.
- Evidence-based biochar selection facilitated excellent long- and short-chain PFAS removal from water.
- Results would be useful in the design of flow-through treatment systems for PFAS removal from water.

**Keywords** Adsorption, Linear model, Prediction, Regeneration, PFAS, Recycled water

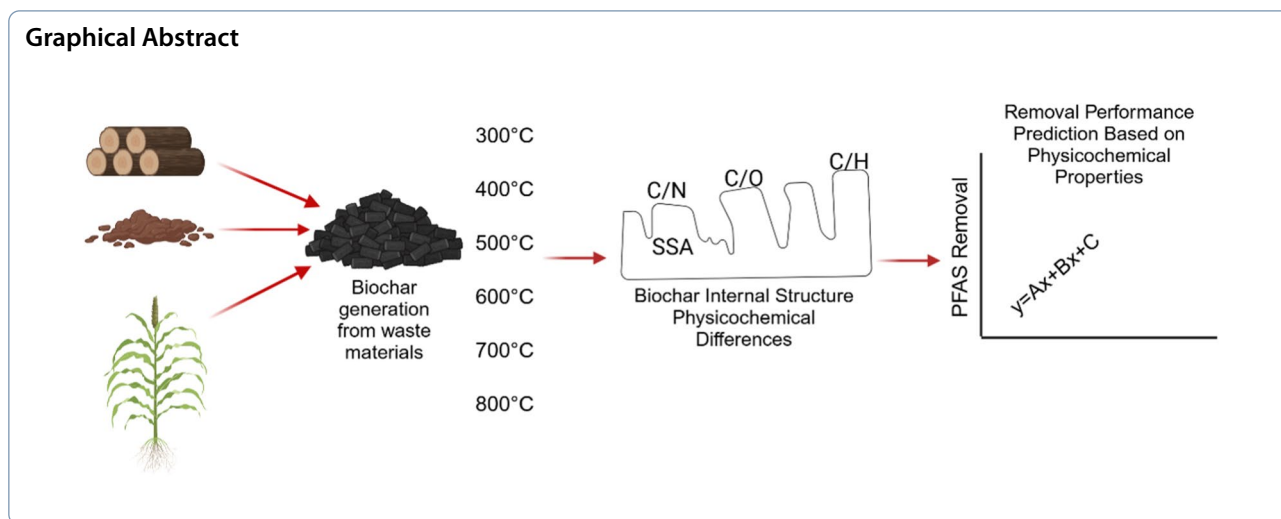
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## 1 Introduction

Per- and polyfluoroalkyl substances (PFASs) have gained increasing attention due to their prevalence in the environment as well as in animals and humans. The United States Environmental Protection Agency (US EPA) has recently implemented actions to mitigate the environmental impact of these compounds. For example, in April 2024, US EPA announced the final National Primary Drinking Water Regulation for six PFASs: perfluorooctanoic acid (PFOA), perfluorooctane sulfonic acid (PFOS), perfluorobutanoic acid (PFBA), perfluorobutanesulfonic acid (PFBS), perfluorobutanesulfonic acid (PFHxS), and perfluorononanoic acid (PFNA) to levels that are 10,000 times lower than previously regulated (EPA 2024). PFAS have also been shown to pose a threat to agricultural resources such as soil, plants, water, and soil fauna (Ramos and Ashworth 2024). PFASs enter agricultural compartments primarily via irrigation with recycled water as well as with the application of biosolids, but other sources include biotransformation of precursors, landfill leachate, and air pollution (Ramos and Ashworth 2024). Finding scalable strategies to remove PFAS from agricultural media is therefore of great importance to protect ecosystem and human health.

Biochar presents a promising solution to addressing PFAS contamination in agriculture (Ramos and Ashworth 2024). Biochar is a carbonaceous material produced via the pyrolysis of organic materials and has been used as an adsorbent for remediation of environmental pollutants including PFASs (Fabregat-Palau et al. 2022; Schmidt et al. 2023; Zhang et al. 2019). Biochar is generally a sustainable alternative to more widespread adsorbents such as granular activated carbon (GAC) because it has beneficial cost and life-cycle considerations (Yaashikaa et al. 2020; Zhang et al. 2019). This is

because it is made from organic waste materials (e.g., plants, wood, manure, and sludge) (Ippolito et al. 2020), and has secondary benefits such as improving soil health and sequestering carbon (Yaashikaa et al. 2020). Previous research has shown that using biochar instead of activated carbon is beneficial from a life-cycle perspective in agricultural settings (Ramos and Ashworth 2024). At present, studies on biochar used for removal of PFAS from aqueous matrices report a wide spectrum of removal efficiencies for both long- and short-chained PFAS, ranging from 30 to 95% for long-chained PFAS and 10 to 92% for short-chained compounds (Behnami et al. 2024; Fabregat-Palau et al. 2022; Inyang et al. 2017; Liu et al. 2021; Nguyen et al. 2023; Steigerwald et al. 2021; Zhang et al. 2021). This divergence in results, as well as the potential environmental benefits of using biochar instead of other adsorbents as a PFAS remediation method in agricultural contexts (Ramos and Ashworth 2024), present the need for a systematic evaluation of the impact of different preparation methods and feedstocks on long- and short-chain PFAS removal by biochar. There is also a growing need to assess viable regeneration strategies for biochar and other adsorbent materials used for PFAS treatment of agricultural media as well as in other contexts. This may allow for an improved comparison of the relative potential of biochar as a widely scalable material for PFAS removal from agricultural compartments compared to other adsorbents.

The aim of this work was to address limitations in the current knowledge of biochar as a potential material for PFAS remediation from aqueous matrices by relating PFAS removal performance of different biochars to multiple physicochemical properties. This work therefore performed an extensive characterization of biochars derived from four feedstocks that are common agricultural waste

sources (i.e., pine wood chips, grass clippings, walnut shells, and cattle manure) pyrolyzed at a range of temperatures (300–800 °C). These preparation temperatures represent a range that is typically used for biochar preparation (Behnami et al. 2024; Chen et al. 2016; Dalameh et al. 2019; Schmidt et al. 2023), as well as conditions that might be easily replicated to prepare biochar from agricultural waste sources on farm. Systematic evaluations of the performance of these biochars as adsorbents for a range of PFASs were then performed. A total of 17 physicochemical properties of 24 biochars were characterized and these were used to determine the main factors influencing the adsorption of short- and long-chained PFASs in batch systems. Principal component analysis allowed an identification of the primary physicochemical characteristics influencing PFAS removal by the different biochars, and linear models were constructed to predict the removal of PFOS and PFBS considering relevant biochar characteristics. This may allow practitioners to more efficiently select adequate commercial biochars given their physicochemical traits, or to adequately prepare biochars from waste sources given the knowledge of the effect of different parameters on characteristics and subsequent removal performance. Adsorption isotherms were constructed and the effect of different background matrices on the removal of several PFAS was evaluated for biochar prepared in house as well as for a commercial biochar that was selected based on its favorable physicochemical traits. A simple thermal treatment was tested as a reactivation strategy for several biochars to evaluate a method for prolonging the lifetime of the adsorbent since this would have beneficial applications when used at larger scales. More broadly, the systematic evaluations performed in this work may help inform efforts to develop batch as well as flow-through filtration systems for PFASs using biochar at larger scales in agricultural settings.

## 2 Materials and methods

### 2.1 Reagents and PFAS standards

Potassium perfluorooctanesulfonate (PFOS salt; 95%) was obtained from Fisher Scientific (Hampton, NH, USA). Perfluorooctanoic acid (PFOA; 96%), perfluorononanoic acid (PFNA; 97%), potassium perfluorohexanesulfonate (PFHxS salt; 95%), tetrabutylammonium perfluorobutanesulfonate (PFBS salt; 98%) and perfluorobutanoic acid (PFBA, 98%) were obtained from Sigma-Aldrich (St. Louis, MO, USA). The corresponding isotopically labeled internal standards, namely perfluoro-*n*-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanoic acid, sodium perfluoro-1-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanesulfonate, and perfluoro-*n*-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]butanoic acid, sodium perfluoro-1-[2,3,4-<sup>13</sup>C<sub>3</sub>]butanesulfonate, sodium perfluoro-1-[1,2,3-<sup>13</sup>C<sub>3</sub>]hexanesulfonate, perfluoro-*n*-[1,2,3,4,5-<sup>13</sup>C<sub>5</sub>]nonanoic acid, were purchased

from Wellington Laboratories (Guelph, Ontario, Canada). These six PFASs were selected based on the EPA's final PFAS National Drinking Water Regulation (EPA 2024); however, most experiments did not include data for PFBA due to analytical complications. Methanol (LC/MS grade) was obtained from Fisher Scientific. For biochar characterization, all reagents used were of reagent grade and used as received without further purification or modification. Solutions and analytical blanks were prepared using deionized ultrapure water (18.2 MΩ cm).

### 2.2 Biochar preparation and characterization

The feedstocks walnut shell (WS), grass clipping (GC), pine wood chip (P), and cattle manure (M) were used to produce biochar using a Carbolite Gero TF1–1200 furnace (Carbolite Gero, Hope, UK). The materials were heated under N<sub>2</sub> flow (1 L min<sup>-1</sup>) from ambient temperature to 300, 400, 500, 600, 700, or 800 °C at a rate of 5 °C min<sup>-1</sup>. Samples were subsequently pyrolyzed at their target temperatures for 3 h and cooled to room temperature. Following pyrolysis, the biochars were ground to pass an 80-mesh sieve (180 μm), prior to use. The biochars were also washed using deionized (DI) water before use in adsorption experiments to remove residual salts and soluble organic species that could influence adsorption. All 24 biochar products were characterized in terms of pH, cation exchange capacity (CEC), C, H, N, O contents, specific surface area (SSA), ash content, volatile content, pore volume, and pore diameter, according to standard techniques as previously described (Schmidt et al. 2023) (See Supporting Information).

One commercial biochar referred to herein as Rogue biochar (Oregon Biochar Solutions, White City, OR USA) was selected according to the biochar characteristics that were found to have a positive effect on PFAS removal performance. The feedstock for this biochar was 80% softwood, 15% hardwood, and 5% nutshells and pyrolyzed at >900 °C in a 15% moisture environment (Valenca et al 2020). Physicochemical characteristics for this biochar can be found in Supporting Information. For validation of the generated linear model, three additional biochars synthesized in house were used, these were date palm leaf biochar pyrolyzed at 800 °C, pistachio shell biochar pyrolyzed at 800 °C, and rice husk biochar pyrolyzed at 400 °C.

### 2.3 Adsorption experiments

PFAS stocks were prepared in 70:30 methanol/DI water at a concentration of 1 g L<sup>-1</sup>. Aliquots from each PFAS stock were added individually to each vial at the target starting concentrations and these were sampled to be analyzed for determination of the starting concentrations. All experiments to evaluate adsorption

characteristics were carried out in 1.5-mL polypropylene microcentrifuge tubes.

To initially determine a suitable adsorbate concentration for subsequent experiments, the performance of biochars prepared from different feedstocks at 600 °C was tested at an initial concentration of 50  $\mu\text{g L}^{-1}$  of each PFAS and at slurry biochar doses of 0.05, 0.1, 0.5, 1, 3, 5, and 10  $\text{g L}^{-1}$  (Figure S1A, S1B). This temperature was selected because it was expected to achieve relatively good PFAS removal compared to biochars generated at lower temperatures based on previous understanding of the effect of temperature on SSA of biochar, so it was taken as a good starting point for comparing the different materials in order to determine the optimal slurry concentration. Based on data from this experiment (PFAS removal at different biochar doses for the different feedstocks and preparation methods), 1  $\text{g L}^{-1}$  was selected as the dose for subsequent adsorption experiments since this dose showed performance differences across the various biochars while still demonstrating relatively high removal of certain compounds. This was deemed important because of our interest in understanding the impact of differing feedstocks and pyrolysis temperatures on PFAS removal.

Equilibrium adsorption was studied using three approaches: (i) PFAS removal performance was studied both individually and in mixtures in DI water at an initial concentration of 50  $\mu\text{g L}^{-1}$  for each PFAS. (ii) Equilibrium adsorption isotherms were assessed over a range of concentrations (5 to 10,000  $\mu\text{g L}^{-1}$ ) in DI water. Adsorption isotherms were obtained for one biochar from each feedstock category based on the previous experiments to determine which biochar would achieve the highest removal for different PFAS. (Figures S4, S5). (iii) Biochar removal performance towards mixed PFAS (50  $\mu\text{g L}^{-1}$ ) was also determined in a background matrix of effluent from a municipal wastewater facility, which had characteristics representative of recycled water used for irrigation. An initial concentration of 50  $\mu\text{g L}^{-1}$  was selected for use in the experiments based on a desired balance between (i) examining adsorption behavior at concentrations considered sufficiently low to be environmentally relevant and (ii) our analytical detection limits when analyzing the post-adsorption solutions. Suspensions were not pH-adjusted and therefore matched the inherent pH of the different biochars. Biochar selection for this experiment included one biochar from each feedstock generated at a low temperature (400 °C) and one generated at a high temperature (800 °C) in order to test biochars with different physicochemical traits since the pyrolysis temperature is known to directly impact these properties. All experimental conditions were tested in triplicate. The initial concentration of 50  $\mu\text{g L}^{-1}$  allowed comparison

with previous studies that have reported adsorption behavior at a range of concentrations less than or equal to 100  $\mu\text{g L}^{-1}$  (Dalameh et al. 2019; Liu et al. 2021; Zhang et al. 2021).

All suspensions of biochars and PFASs were shaken for 24 h, which is a length of time that has been shown to be adequate to reach equilibrium in PFAS adsorption experiments using biochar (Fabregat-Palau et al. 2022). The pH values of the suspensions were neither adjusted nor buffered so as to understand the adsorption behavior of the different biochars without further controls because of the intended applications of these materials in simple large-scale flow-through systems. The pH values of all biochar suspensions were within the range of 6.5–7.1. Following this equilibration period, samples were withdrawn, centrifuged at 14,000 rpm for 15 min and the supernatant passed through a 0.2  $\mu\text{m}$  Nylon syringe filter into a polypropylene vial before liquid chromatography–tandem mass spectrometer (LC–MS/MS) analysis. All studies were performed in triplicate ( $n=3$ ). Adsorption was quantified as the difference between the initial and final amounts of all tested PFASs. Negative controls were assessed for all experiments in triplicate.

#### 2.4 Regeneration of saturated biochar

Post-pyrolysis air oxidation was tested as a strategy for biochar regeneration according to previous studies (Alin-ezhad et al. 2024; Wang et al. 2023; Xiao et al. 2016; Yang et al. 2021). Biochars from each feedstock prepared at 800 °C were equilibrated with PFAS as detailed above but with a starting concentration of 1  $\text{mg L}^{-1}$  and in 50 mL polypropylene tubes (instead of 1 mL) to produce larger masses of biochar to be used for the subsequent experiments. This higher starting concentration was selected because of the focus on reactivation, stipulating that if an effect were observed at this high concentration, a similar regeneration could be expected at lower environmental concentrations. Following equilibration, supernatant samples were aliquoted, and then prepared as detailed above. Biochars were retrieved and dried at room temperature before reactivation. For reactivation, biochars were placed in closed ceramic crucibles in a furnace at either 200, 300, or 400 °C for 30 min. All experimental conditions were tested in triplicate. After cooling back to room temperature, these biochars were used for a subsequent round of adsorption experiments following the methodology detailed above.

PFASs were extracted from a subset of the saturated and thermally treated biochars to assess any impacts of the thermal treatment on decomposition of the PFAS compounds. The procedure for PFAS recovery from saturated adsorbents has been reported and evaluated previously (Wang et al. 2023; Xiao et al. 2020). Briefly,

saturated biochars were dried at room temperature and weighed to ensure equal mass following thermal treatment. Extraction was performed with 10 mL of methanol amended with 100 mmol L<sup>-1</sup> of ammonium acetate and the samples were sonicated at 60 Hz for 30 min in an ultrasound bath (Sonic Systems, Inc. Newton, PA). Negative controls included samples of biochar that had not been previously saturated with PFAS and were assessed in triplicate. Samples were then prepared for PFAS analysis as detailed below.

## 2.5 PFAS analysis

Stock solutions of isotopically labeled internal PFAS standard were prepared using high-purity methanol in a 70:30 ratio of methanol to DI water. The internal standard of each compound was added to a concentration of 25 µg L<sup>-1</sup> for PFOS, PFOA, PFHxS, and PFNA and 50 µg L<sup>-1</sup> for PFBA and PFBS according to previous chromatography method optimization. Samples were prepared in 200 µL polypropylene vials and thoroughly mixed. Analyses were performed using an Agilent 1290 Infinity II HPLC coupled with an Agilent 6470B triple quadrupole (QQQ) mass detector equipped with an Agilent Jet Stream (AJS) source. Further details are available in Supporting Information.

## 2.6 Data analysis

Removal efficiency (RE), sometimes also referred to as percent removal, was calculated by:

$$RE (\%) = \left( \frac{C_0 - C_e}{C_0} \right) * 100$$

where  $C_0$  is the initial PFAS concentration in solution (µg L<sup>-1</sup>) and  $C_e$  is the final concentration after 24 h of equilibration (µg L<sup>-1</sup>).

The solid–liquid partitioning coefficient,  $K_d$  (L kg<sup>-1</sup>) was defined as:

$$K_d = \frac{C_s}{C_e}$$

where  $C_s$  is defined as:

$$C_s = \frac{(C_0 - C_e) * V}{m}$$

$V$  (mL) is the volume of the solution and  $m$  (g) is the dry mass of biochar added to each reaction vessel.  $C_s$  here describes the mass of PFAS adsorbed per mass of adsorbent, and is thus equivalent to the parameter  $q_e$  in this context.

Adsorption isotherms were fitted using the PUPAIM package in R statistical programming language as well as the package `minpack.lm` (RStudio Version 2023.9.1.494).

Principal component analysis (PCA) was performed using the R packages `FactoMineR` and `factoextra` (RStudio Version 2023.9.1.494) to identify the key biochar properties affecting PFAS sorption. To predict the removal of PFOS or PFBS, linear regression models were fitted by two methods and compared. First, the most influential variables (the highest contribution to the principal components) as depicted by PCA were used to construct a linear model by iteratively removing variables until a suitable fit was attained (as determined by  $R^2$ , root mean squared error [RMSE], and residuals). A second approach to model construction involved stepwise multiple linear regression using the R packages `MASS`, `tidyverse`, `leaps`, and `caret` (RStudio Version 2023.9.1.494) which accounts for multicollinearity. This second approach was deemed superior and was therefore the model that was presented below. These functions consider the  $p$ -value for each included parameter in terms of improving the overall model fit. When depicting variables involving ratios of two elements present in the different biochars, molar ratios are used.

## 3 Results and discussion

### 3.1 Effect of biochar properties on PFAS adsorption

In general, long chained compounds (PFOS, PFOA, PFNA) were removed more effectively than short chained PFAS (PFBS, PFBA, and PFHxS) by biochars prepared from grass clippings, manure, pine wood chips, and walnut shells at a pyrolysis temperature of 600 °C (Figure S2) when tested in mixed PFAS systems at a starting concentration of 50 µg L<sup>-1</sup>. PFOS (C8) was very effectively removed (>90%) by biochars derived from all feedstocks, while the C8 carboxylate compound PFOA was less successfully retained (16–66%). This is consistent with previous studies on biochar removal of PFASs since biochar removes PFASs primarily via hydrophobic interactions and these are stronger in sulfonate compounds compared to carboxylates of the same chain length (Gagliano et al. 2020; Inyang et al. 2017). Similarly, due to the positive correlation between PFAS chain length and hydrophobicity, PFNA (C9) was more successfully removed compared to PFOA. Removal of PFNA was high and not significantly different between pine, manure, and walnut shell biochars, while removal by biochar synthesized from grass clippings was much lower, which may be related to the  $N_2$  surface area of this biochar compared to the others (Table 1). Short chained compounds were removed between 15–65% for PFHxS (C6), 1–61% for PFBS (C4), and 0–55% for PFBA (C4). The lowest removals of PFBA, PFBS, and PFHxS were observed for pine biochar, while removals by the other three biochars were not statistically different from each other. This lower RE accords with previous studies on adsorption of PFAS by carbonaceous

**Table 1** Physicochemical properties of biochars used in this study

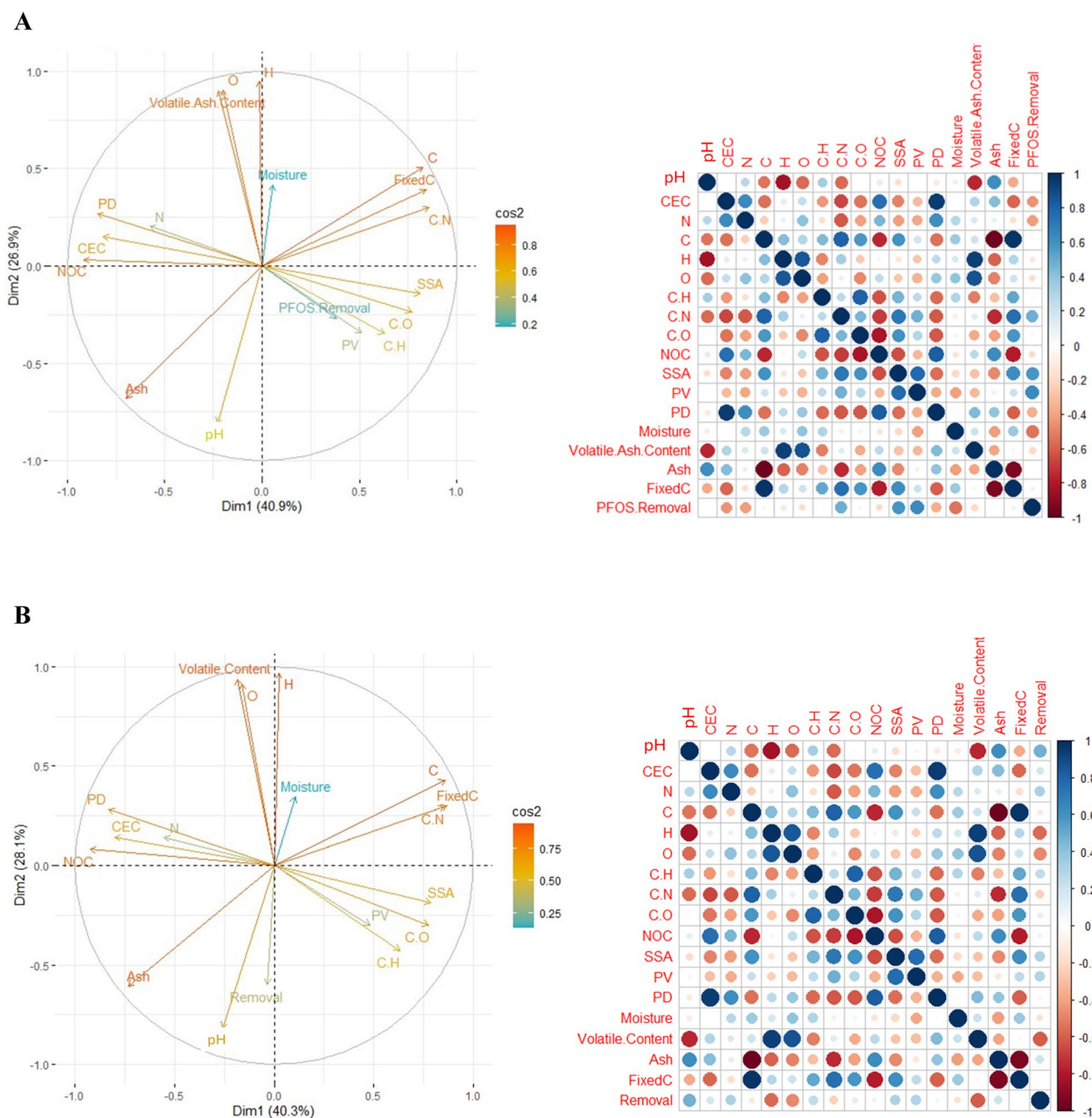
Biochar	pH	CEC	%N	%C	%H	%O	SSA	PV	PD	Moisture	Volatile Cont	Ash	C
GC 300°	8.57	19.3	3.42	51.1	5.0	22.3	0.93	0.006	24.99	2.05	19.94	29.35	47.32
GC 400°	9.6	16.12	3.00	49.4	3.2	17.3	2.54	0.009	14.15	1.02	13.24	29.35	47.32
GC 500°	10.6	4.1	2.72	49.1	2.1	14.3	5.97	0.002	9.94	2.06	11.05	32.59	47.48
GC 600°	11.0	4.0	2.31	47.4	1.3	13.4	6.81	0.002	9.48	2.52	13.92	33.56	53.20
GC 700°	11.3	6.0	2.36	51.4	0.8	14.1	15.14	0.024	6.22	2.89	17.72	35.10	53.85
GC 800°	10.8	9.5	2.40	51.0	0.7	14.8	9.29	0.018	7.88	5.10	13.92	34.78	51.31
M 300°	9.0	18.2	1.00	11.8	1.3	14.9	2.83	0.015	21.18	1.38	17.72	81.50	0.78
M 400°	9.7	11.7	0.63	8.3	0.6	8.7	5.55	0.021	15.31	1.10	13.29	86.32	0.38
M 500°	10.1	9.0	0.62	8.7	0.5	8.3	8.17	0.022	10.63	0.90	12.22	87.36	0.42
M 600°	11.3	4.4	0.50	8.1	0.4	8.3	21.33	0.032	6.03	0.73	10.69	88.66	0.65
M 700°	11.8	3.7	0.35	7.6	0.2	6.4	32.97	0.038	4.56	0.73	9.18	88.73	2.09
M 800°	11.5	2.6	0.29	7.6	0.2	6.7	30.89	0.035	4.48	0.72	8.29	91.45	0.26
WS 300°	6.8	9.0	0.40	67.1	5.1	26.9	1.38	0.005	12.89	3.49	52.55	2.33	45.12
WS 400°	8.9	2.2	0.33	78.8	3.5	16.1	7.26	0.008	4.36	3.61	29.15	2.10	68.75
WS 500°	8.7	0.5	0.33	87.2	3.0	10.4	118.44	0.040	1.34	2.21	17.68	2.34	79.98
WS 600°	8.9	0.5	0.30	89.8	2.1	7.0	410.96	–	1.25	2.38	15.68	2.40	81.92
WS 700°	9.6	1.0	0.33	91.8	1.1	5.8	142.07	0.005	1.27	2.61	12.15	2.85	84.99
WS 800°	10.0	1.3	0.43	91.9	0.7	5.7	185.03	–	0.85	2.86	10.17	3.07	86.76
P 300°	7.7	0.9	0.18	74.6	5.3	20.6	2.49	0.003	5.04	1.30	46.02	0.70	53.28
P 400°	7.4	0.6	0.18	79.0	4.4	15.6	1.18	0.002	5.89	1.71	31.34	0.95	67.71
P 500°	8.0	0.4	0.20	87.5	2.3	6.8	645.48	0.292	1.34	1.19	20.01	1.04	78.96
P 600°	9.0	0.5	0.21	88.7	2.3	5.6	457.34	0.187	1.64	0.91	12.76	1.20	86.03
P 700°	9.7	0.6	0.16	86.8	1.3	4.4	465.48	0.250	1.58	1.24	11.90	1.21	86.89
P 800°	9.8	0.6	0.24	94.3	0.8	3.9	570.22	0.237	1.13	1.54	11.02	1.33	87.65

GC Grass Clippings, M Manure, WS Walnut shell, P Pine wood chips. All temperatures are in °C. C represents fixed carbon. Pore volume (PV) is given in  $\text{cm}^3 \text{g}^{-1}$ ; pore diameter (PD) in nm; cation exchange capacity (CEC) in  $\text{cmol charge kg}^{-1}$ ; specific surface area (SSA) by  $\text{N}_2$  adsorption in  $\text{m}^2 \text{g}^{-1}$ ; whereas moisture, volatile, ash, and fixed carbon contents are given as percentages (%)

materials since shorter compounds interact less favorably with the hydrophobic biochar surface (Gagliano et al. 2020; Inyang et al. 2017). The wide range of removal performances corresponds to what has been observed previously in the literature (Behnami et al. 2024; Inyang et al. 2017; Liu et al. 2021), and further emphasizes the need for a systematic evaluation of PFAS removal by biochar to facilitate adequate selection of feedstocks and preparation methods to make biochar a viable strategy for large-scale treatment of PFAS-contaminated waters.

Biochar characterization results are shown in Table 1. These data depict the impact of preparation temperature and feedstock on the physicochemical properties of biochar. These effects have been detailed previously (Behnami et al. 2024; Ippolito et al. 2020; Ramos and Ashworth 2024; Schmidt et al. 2023), so the focus herein is on the effect of these characteristics on the adsorption of short- and long-chain PFAS. Principal component analyses were performed and heatmaps were generated to explore the relationships between the different characteristics of the 24 biochars prepared from four feedstocks at six temperatures and the removal of one long-chained

(PFOS) and one short-chained (PFBS) compound (Fig. 1). These assessments were performed based on experiments at a starting concentration of  $50 \mu\text{g L}^{-1}$  in systems of mixed PFBS, PFOS, PFOA to compare one carboxylate and one sulfonate compound as well as compare short- and long-chained compounds. There were two missing values in Table 1, which the authors were unable to attain, so it is important to acknowledge that, though minor, omission of these data may have influenced the PCA and models derived. Results from PCA and correlation analyses showed that for PFOS, physicochemical biochar properties influencing removal performance included C, H, O, and their ratios as well as SSA and pore volume. For PFBS, influencing factors were C, N, H, O and their ratios as well as SSA and moisture content. The relevance of SSA, C/N, and C/O is consistent with a previous study by the present authors, which used data available from the literature to determine the main factors influencing PFAS sorption to several biochars (Ramos and Ashworth 2024), as well as with similar studies by Fabregat-Palau et al. (2022) and Krebsbach et al. (2023) on removal of PFOS by carbonaceous materials. Increases in SSA increase



**Fig. 1** Principal Component Analysis (PCA) and heat maps for removal of (A) PFOS and (B) PFBS by 24 biochars considering 12 physicochemical properties. Depicted NOC represents the ratio (N + O)/C

PFAS removal because of the greater number of sites available for adsorption (Gagliano et al. 2020; Krebsbach et al. 2023; Schmidt et al. 2023). The C/O ratio is known to be related to hydrophobicity, with higher values resulting from the loss of oxygen-containing functional groups (Fabregat-Palau et al. 2022). This ratio has also been shown to be a good indicator of aromaticity of a material (Fabregat-Palau et al. 2022). This is consistent with previous findings suggesting that basic sites in  $\pi$ -electron-rich

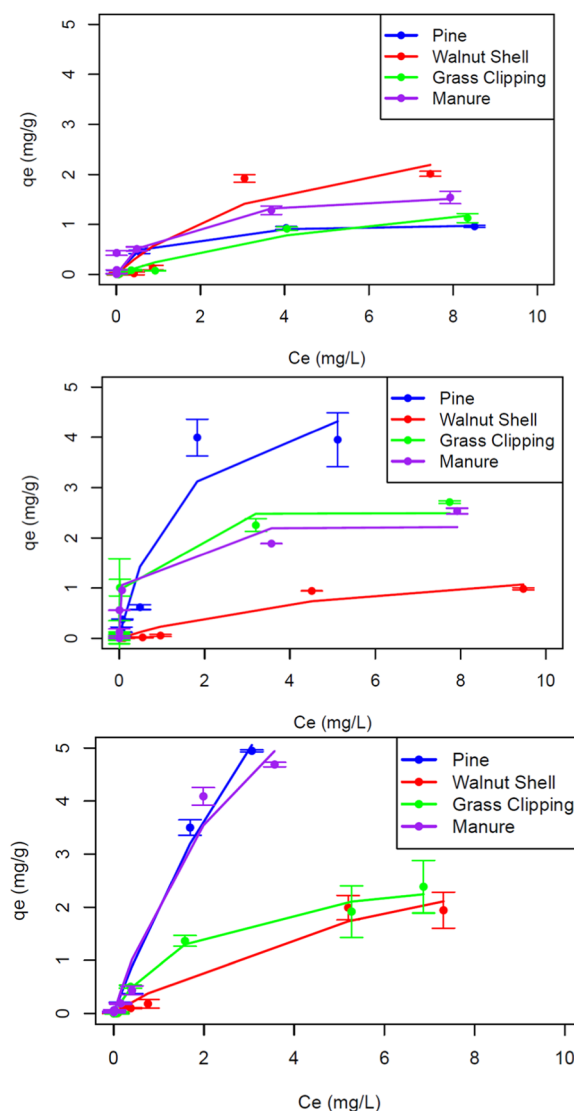
regions in biochar are important for PFAS sorption (Fabregat-Palau et al. 2022). The ratio of (N + O)/C has been suggested as an indicator of the polarity of a surface, so a negative correlation between this value and PFBS removal is consistent with the primary PFAS removal mechanism, i.e., hydrophobic interaction (Gagliano et al. 2020; Valenca et al. 2020). The pH value of the biochar suspensions was seen to be relatively significant in determining the removal of PFBS, with higher pH values

resulting in lower PFBS removal. Since experimental pH values were all circumneutral, the effect might be due to differences in the surface charge of the different biochars. It is noteworthy that there are differences between the variables deemed important by Pearson’s coefficients shown in the heat map and those presented by PCA. This is because PCA considers collinearities between the variables, which is not the case in the Pearson correlation analyses. It is likely that the reduction in dimensionality performed by PCA removed important components that can explain the strong relationships shown in the heat-maps between PFAS removal and physicochemical traits (Fig. 1).

Further studies are required to further characterize the surface charges as well as to evaluate the impact of altering solution pH on PFBS removal by different biochars. Additionally, consideration of dissolved organic carbon (DOC), and anion exchange capacity variations between the biochars might provide additional value to the model so consideration of these parameters may be beneficial. As noted, the relevant characteristics are all directly related to the strategy used to generate biochar (i.e., pyrolysis temperature and feedstock used), so this understanding may allow practitioners to select suitable preparation methods depending on the desired outcome for different PFAS.

### 3.2 Development of linear model to predict PFAS removal

A linear model was constructed based on these findings to predict the removal of PFOS and PFBS given the physicochemical properties of biochar. These compounds were selected to test prediction performance for one short-chained and one long-chained compound. Fabregat-Palau (2022), as well as the present authors (Ramos and Ashworth 2024), previously constructed linear models that contained a term for the PFAS chain length to make the prediction widely applicable for a range of PFAS. This was also attempted in this work, however no models accurately fit the data. This is likely due to insufficient data since only two chain lengths were considered, and the overall sample size (n=24) was much smaller than those used in previous studies (n=80–90). Since the present study considered a much larger selection of physicochemical biochar properties compared to what has been reported for other biochars, it was not possible to expand the experimental dataset from literature data. As a result, one model was constructed for each sulfonate chain length herein. Validation of the models using a wider range of PFAS chain lengths could allow for the construction of a more generalizable model, and inclusion of additional data from more biochars could also improve the predictive power of these models. Machine learning may also be used to improve the predictive



**Fig. 2** Langmuir isotherms for the removal of PFOA (top), PFOS (middle), PFBS (bottom) by biochar pyrolyzed at different temperatures made from four feedstocks. The pyrolysis temperatures were: For PFOA removal W 400 °C, GC 600 °C, P 600 °C, M, 400 °C; for PFOS W 800 °C, GC 800 °C, P 800 °C, M 800 °C; for PFBS W 400 °C, GC 800 °C, P 800 °C, M 400 °C. These temperatures were selected based on preliminary studies identifying greatest removal for the different compounds by the various biochars (Figure S4)

performance of this and similar models (Cheng et al. 2023). Additional statistics are provided in supporting information (Figure S3).

$$\text{PFOS Removal} = 2.0 \cdot N - 0.4 \cdot C + 0.1 \cdot C/N + 0.03 \cdot \text{SSA} + 90.$$

$$R^2 = 0.90, p < < 0.01, n = 24.$$

$$\text{PFBS Removal} = 13 \cdot N + -0.9 \cdot C - 4.9 \cdot O - 0.05 \cdot C/N - 1.6 \cdot C/O + 111 \cdot (N + O)/C + 0.05 \cdot \text{SSA}.$$

$$R^2 = 0.91, p < < 0.01, n = 24.$$

**Table 2** Predicted vs. experimental performance for PFBS and PFOS by Rogue, date palm leaf, pistachio shell and rice husk biochars

Biochar feedstock & pyrolysis temp	Predicted removal PFOS (%)	Experimental removal PFOS (%)	Predicted removal PFBS (%)	Experimental removal PFBS (%)
Rogue (80% softwood, 15% hardwood, and 5% nutshells) > 900 °C	86	98 ± 1	90	86 ± 1
Date palm leaf 800 °C	75	81 ± 15	77	68 ± 9
Pistachio shell 800 °C	65	55 ± 11	63	71 ± 14
Rice husk 400 °C	70	65 ± 23	62	77 ± 17

Predicted values were calculated based on the linear model generated presented above. Uncertainties in the experimental removal of PFOS and PFBS represent the standard deviation from experimental triplicates

Stepwise regression was determined to be a superior approach over using the data from PCA because when comparing the statistical fits of the two models, the fits obtained when using the parameters determined by PCA were poor (lower  $R^2$ , poor performance when evaluating residuals, higher p-values, higher root mean square error). It is noteworthy in the model for PFOS that some of the parameters that were determined as important in the PCA related to surface hydrophobicity did not appear in the model following stepwise regression (notably C/O, C/H). However previous mechanistic investigations have found that nitrogen as well as the C/N ratio have the largest impact on removal of PFOS out of all measured elements and elemental ratios (Krebsbach et al. 2023). This is because the positively charged N-containing groups of biochars have been reported to strongly sorb to negatively charged PFAS via electrostatic interactions. Biochars with high N content have repeatedly shown high PFOS removal (Krebsbach et al. 2023). Given the framework for predictive removal of PFAS based on biochar characteristics presented above, machine learning may be a promising tool for further investigation into the predictive removal by different biochar materials (Cheng et al. 2023).

To test the performance of these models beyond the statistics, four additional biochars were tested for their performance at removing PFOS and PFBS. The physicochemical properties of these biochars are presented in supporting information (Table S5). Given the characteristics and the generated models, the removal performance predicted and experimental values for the four biochars are presented below (Table 2).

In general, the predicted values fell within the observed experimental errors in most cases. The exception was the Rogue biochar, where the experimental values obtained for PFOS removal were higher than those of the predicted removal. It is possible that this is partially the result of the SSA (one of the parameters used in the predictor equation for PFOS) of Rogue biochar. The SSA of

Rogue biochar was relatively high, whereas in the data used to generate the predictive model only two values were larger and the majority were orders of magnitude smaller. This might negatively impact the predictive power of the model for biochars with high SSA. Further studies providing a larger dataset might improve the fit for this biochar, and the set of equations provided might be best suited to predict the performance of biochars with characteristics that are within the ranges of those used to generate the model.

It is of significant note that the removal attained by Rogue biochar for PFBS (86% from an initial concentration of  $50 \mu\text{g L}^{-1}$ ) was larger than what has been reported for most biochars for this short-chained compound (i.e., <20% for wood waste biochar at a starting concentration of  $1000 \mu\text{g L}^{-1}$ , 60% for pine-spruce wood biochar at a starting concentration  $3\text{--}5 \mu\text{g L}^{-1}$ ) (Dalameh et al. 2019; Zhang et al. 2021), with a performance comparable to the biochar with the highest previously-reported capacities for short-chained PFAS removal (92–96% at a starting concentration of  $1 \mu\text{g L}^{-1}$  and 80–89% at a starting concentration of  $100 \mu\text{g L}^{-1}$ ) (Liu et al. 2021). This biochar is also superior to most reports for granular activated carbon (70% for a starting concentration of  $1 \mu\text{g L}^{-1}$  and 45% for a starting concentration of  $100 \mu\text{g L}^{-1}$ ) (Gagliano et al. 2020). This illustrates the potential for certain biochars to be used in the remediation of both short and long chain PFAS in agricultural contexts if feedstock and pyrolysis temperature are carefully selected to produce preferable biochar characteristics.

### 3.3 Adsorption isotherms

Adsorption isotherms for PFOA, PFOS, and PFBS were fitted for a selection of the 24 biochars that were previously found to achieve the greatest removal for each compound and respective feedstock (Fig. 2). Non-linear Langmuir models were fit to the data and are shown here since non-linear isotherm models have been widely used by others to fit adsorption data for PFAS on

biochar (Inyang et al. 2017; Liu et al. 2021; Steigerwald et al. 2021; Zhang et al. 2021). The greatest adsorption capacities were observed for PFOS (1–6 mg g<sup>-1</sup>), followed by PFOA (1–3 mg g<sup>-1</sup>) and PFBS (1–4 mg g<sup>-1</sup>). Adsorption affinities followed the same trend, with the highest affinities observed for PFOS ( $K_L = 0.2\text{--}14$  L mg<sup>-1</sup>) followed by PFOA ( $K_L = 0.1\text{--}2$  L mg<sup>-1</sup>) and PFBS ( $K_L = 0.3\text{--}0.5$  L mg<sup>-1</sup>). This is consistent with previous observations of PFAS adsorption capacity by other adsorbents as well as with the superior removal performance for long-chained sulfonates compared to carboxylates and for long-chained compounds compared to short-chained (Fabregat-Palau et al. 2022; Gagliano et al. 2020).  $K_L$  values for these biochars are comparable, or superior, to those reported by Liu et al. (2021), who reported a reed-straw derived biochar with the highest yet recorded PFAS removal potential.

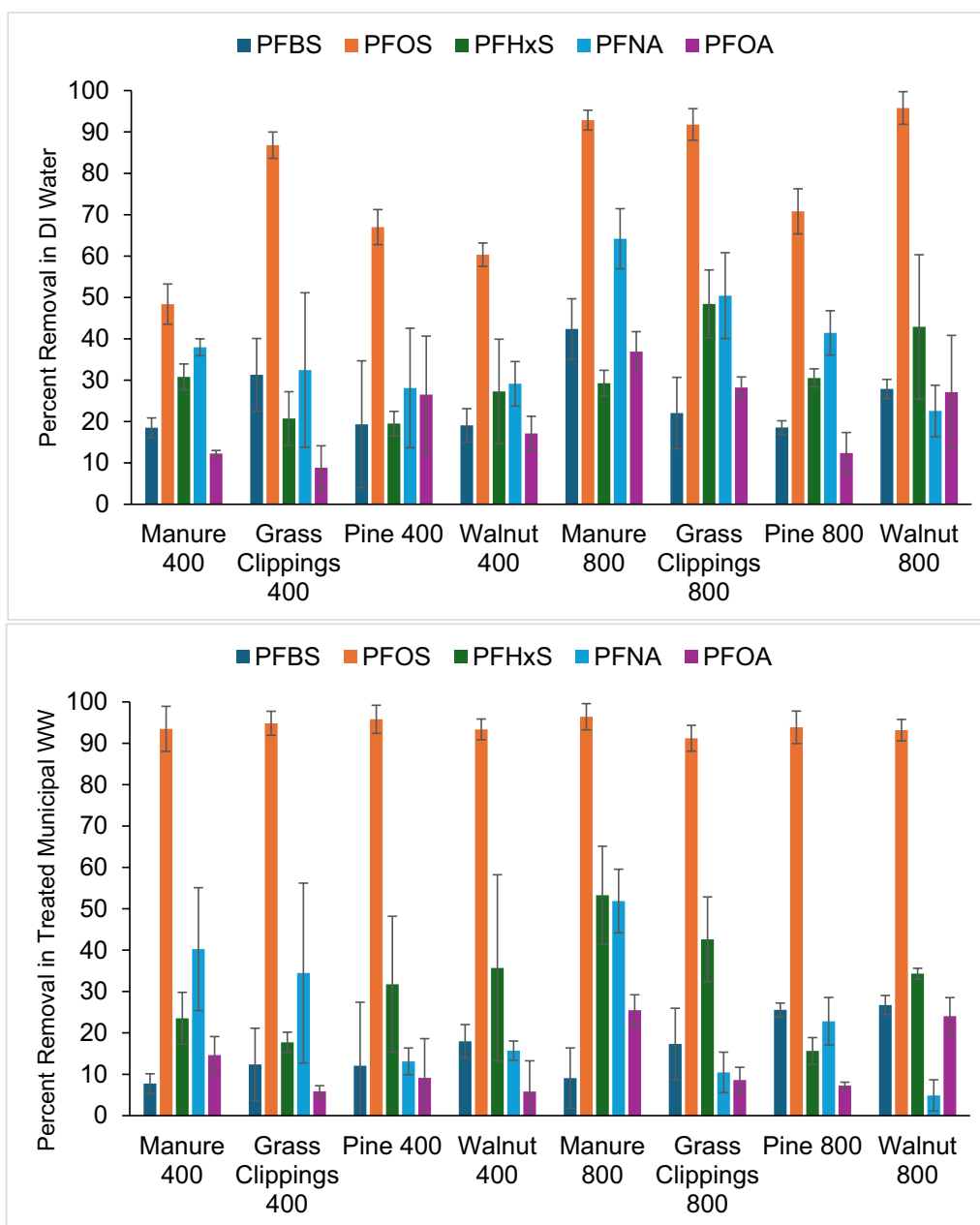
#### 3.4 Effect of background matrix on removal performance

The removal efficiencies (percent removal) for mixed solutions of PFBS, PFOS, PFOA, PFHxS, and PFNA under different background matrices (deionized water [DI] or treated municipal wastewater) are shown in Fig. 3A, B. Water chemistry characteristics of the treated municipal wastewater can be found in Supporting Information (Table S4). For biochars generated in house, the removal performance decreased in treated municipal wastewater compared to DI water. This is consistent with previous observations that the ions prevalent in treated wastewater, as well as DOC, can decrease the PFAS adsorption capacity and performance of carbonaceous adsorbents due to pore-blockage effects (Gagliano et al. 2020; Nguyen et al. 2020; Yu et al. 2012). The decrease in RE was almost non-existent in the case of PFOS, which is likely due to the strong hydrophobic interactions that occur between the biochar surface and this compound (Gagliano et al. 2020). However, as shown in Fig. 4, adequate selection of the physicochemical properties of a biochar (e.g., Rogue biochar) can also mitigate the effects of competition between PFAS in mixed systems as well as the competition effects existing in more complex water chemistries. This is likely due to the high surface area of the Rogue biochar as well as its favorable elemental ratios, as discussed above. It is noteworthy that the removal efficiencies for all tested PFAS by Rogue biochar in treated municipal wastewater are superior to those of most present studies discussing PFAS removal by biochar in similar water matrices, pointing to the potential applicability of using this material (and materials with similar physicochemical properties) for PFAS remediation in reused water, even for short-chained compounds.

#### 3.5 Regeneration using thermal reactivation

Thermal reactivation in the presence of oxygen was tested as a strategy to regenerate the biochars based on previous work identifying this as a viable strategy for biochar regeneration (Wang et al. 2023; Xiao et al. 2016; Yang et al. 2021). In this case, regeneration refers to the recovery of some of the adsorption capacity of the adsorbent material following full or partial saturation with PFAS to be able to use the material over subsequent saturation cycles. Previous work has found that PFAS adsorbed to GAC and soil can melt, evaporate, or decompose by heating above a threshold temperature, and low temperature desorption has been applied as a reactivation approach for GAC and biochar (Alinezhad et al. 2024). Decomposition of PFAS has been observed to initiate at temperatures as low as 200 °C when bound to adsorbents like GAC, with mineralization occurring at higher temperatures (above 700 °C) (Alinezhad et al. 2024; Wang et al. 2023). PFASs were extracted from saturated biochars before and after treatment at 200 °C and 400 °C (Figure S6) and this was used to assess the impact of different treatment temperatures on the presence of PFOS and PFBS remaining on the adsorbent surface. For all tested biochars (pine, walnut, grass, manure, and Rogue biochar) PFOS was almost completely retained on the surface when the biochars were treated at 200 °C, but the value decreased in all cases when treated at 400 °C. The relative presence of PFBS on the surface of the biochars following treatment at 200 °C and 400 °C was lower in all cases, pointing to lower required energy for the desorption of this compound or for its decomposition. This is consistent with weaker interactions between biochar and short-chained PFAS compared to long-chained compounds.

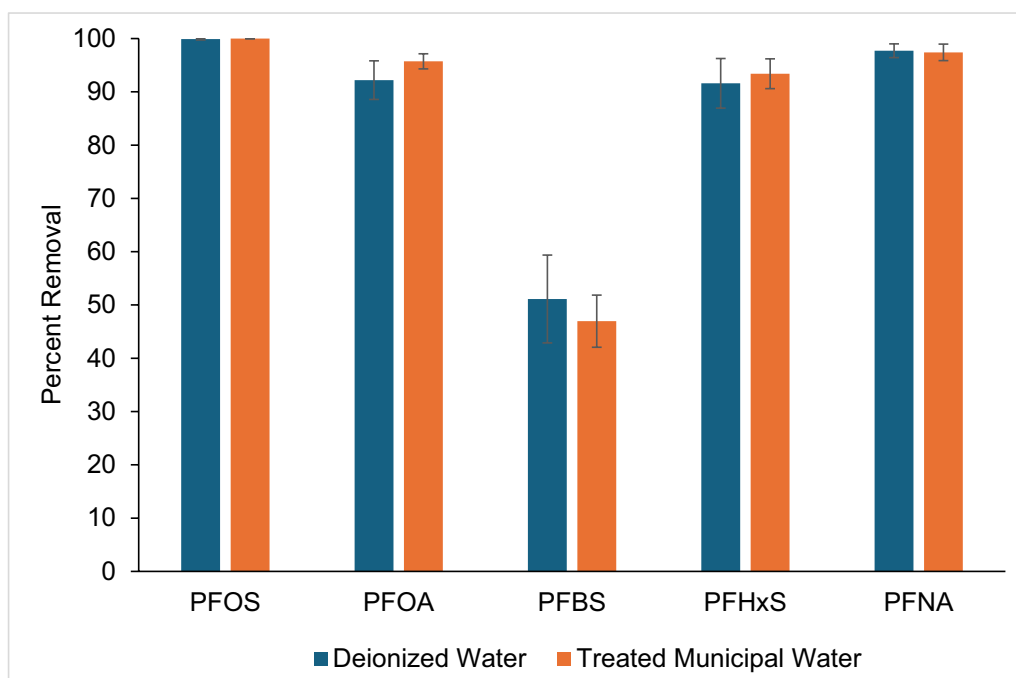
Low temperature thermal treatment can be used to reactivate biochar over subsequent cycles (Fig. 5) with slightly larger effects at higher temperatures. The significance of the potential reactivation at temperatures as low as 200–400 °C is that this is a simple and non-intensive strategy to potentially increase the lifetime of biochars by using equipment that can realistically be used in wide scale applications (e.g. in agricultural contexts). Moreover, thermal treatment in the presence of air was observed to increase the PFAS removal efficiency of biochar in many cases (Fig. 5). This is consistent with previous observations that brief periods of thermal treatment in the presence of air following the pyrolysis process improves the SSA and porosity of biochar due to the oxidative removal of tarry deposits or other organic matter present in the pore walls generated during pyrolysis (Wang et al. 2023; Xiao et al. 2016; Yang et al. 2021). Additionally, previous work has suggested that improved PFAS



**Fig. 3** Removal performance for different biochars in deionized water (top) and treated municipal wastewater (bottom). Experiments were performed in mixed systems containing 50 µg/L PFBS, PFOS, PFHxS, PFNA, PFOA. Removal efficiency was detrimentally affected in all cases in treated municipal wastewater compared to deionized water

removal capacity following these air oxidation processes is due to changes in different elemental ratios (i.e. increasing C/O and C/H ratios) resulting in materials with greater hydrophobicity and condensed aromatic structure (Wang et al. 2023; Yang et al. 2021). Treatment at 400 °C improved the removal performance for PFBS of all biochars generated in-house as well as for the commercial Rogue biochar. A previous

study found significant improvements in the retention of long-chained PFAS compared to short-chained (Wang et al. 2023); however, this was not observed in the present study as the improvement was most significant for PFBS as opposed to PFOS. This is likely because PFOS was already removed at 85–99% for all biochars while removal of PFBS before air oxidation was much lower. The impact of this treatment



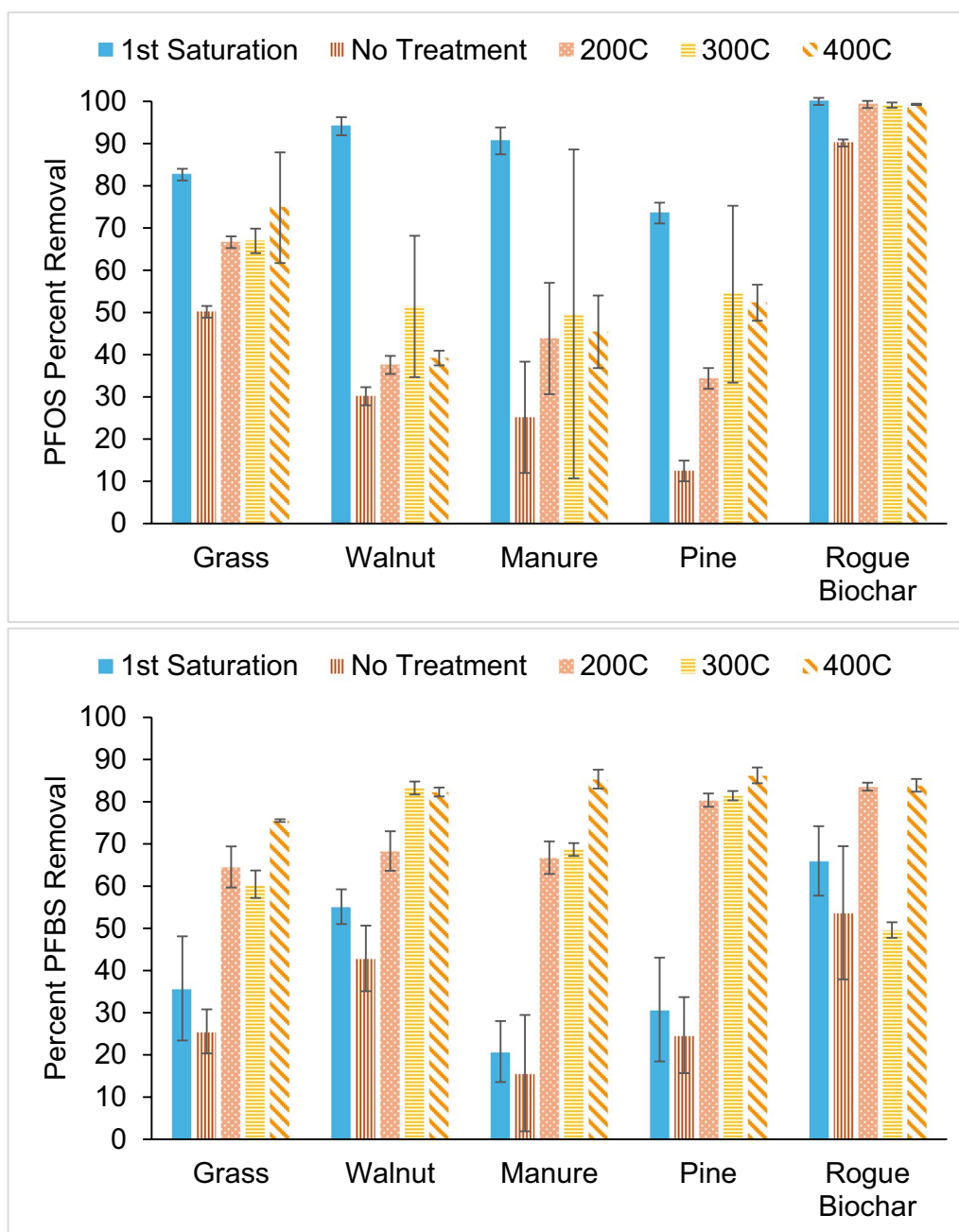
**Fig. 4** PFAS removal performance of commercial Rogue biochar in deionized water (blue) and treated municipal wastewater (orange). Rogue biochar achieved higher removal of all PFAS compared to biochars synthesized in-house. The presence of competing ions in treated municipal wastewater had a negligible effect on the removal efficiency for all biochars in this case. Experiments were performed in mixed systems containing 50 µg/L PFBS, PFOS, PFHxS, PFNA, PFOA

was seen to vary depending on the tested biochar, likely owing to the inherent differences in their physicochemical properties and the role of these in PFAS adsorption, as described above. For instance, the greatest improvements were observed for manure biochar at 400 °C, which showed about five times improved removal efficiency compared to zero to two times for the other biochars (Fig. 5). Manure biochar pyrolyzed at 800 °C had a relatively low SSA (30.9 m<sup>2</sup> g<sup>-1</sup>) as well as low C/O ratio compared to grass clippings, walnut shell, and pine biochar (1.1 vs. 3.4, 16.0, 24.2 respectively). As a result, it is suggested that the changes in C/O and SSA caused by thermal treatment in air could have been the most significant for this biochar. Following thermal treatment at 400 °C, all tested biochars removed >80% PFBS in mixed PFOS/PFBS systems, and Rogue biochar achieved >98% removal of PFOS as well as 85% removal of PFBS, which is superior to all previous biochar studies for PFAS removal. Overall, this strategy appears effective at increasing PFAS removal (particularly for short-chained PFBS) and has potential as a simple post-pyrolysis modification that can be easily carried out and scaled up across diverse settings due to its relatively low energy requirements and low complexity. Further studies are required

to better understand the mechanisms behind the observed results.

#### 4 Conclusion

This work showed that agricultural byproducts may be converted into biochars with different physicochemical properties which provide them with varied functionalities. Evaluation of the primary factors influencing adsorption of long- and short-chained PFASs showed that contrary to what is often shown in the literature, biochar is a viable adsorbent for remediation of a range of such PFAS. Several influential surface characteristics were identified, namely SSA and the ratios of C, N, and O. Appropriate biochar selection (or the feedstock and preparation method used) is imperative to provide optimal physicochemical traits, since these lead to large variations in the success of PFAS removal. As shown in this study through the evaluation of the commercial Rogue biochar, this comprehensive evaluation may allow for future selection of commercial biochars or preparation methods with some confidence of the predicted observed PFAS removal performance by consideration of easily measurable properties. As seen with the Rogue biochar in this study, the performance of successful biochars may be retained even in water chemistries that are



**Fig. 5** Effect of removal performance for PFOS (top) and PFBS (bottom) following simple thermal treatment at 200, 300, or 400 °C. Removal performance after one cycle of saturation followed by thermal treatment improved when biochars were treated at 400 °C. Removal performance of PFBS improved following thermal treatment in most cases

relevant to applications such as agricultural irrigation (e.g., in treated municipal wastewater), attaining >90% removal for PFOS, PFNA, PFHxS, PFOA and >50% for PFBS. Moreover, strategies such as thermal treatment can be used to retain or improve biochar capacity over multiple uses especially in the case of short-chain compounds such as PFBS. Further studies are required to

provide greater validation to the generated linear model, and machine learning may also be a valuable tool to assess the performance of various biochars. Further studies are also required to understand the potential of biochar in a wider range of solution conditions that might be relevant in large-scale applications for treating irrigation water (presence of dissolved organic matter,

influence of multivalent cations). Overall, the presented work provides a framework for further studies developing biochar as a material to be used in treatment of irrigation water for PFAS removal, and the findings provide a basis for more efficient material selection in treatment evaluations.

## Supplementary Information

The online version contains supplementary material available at <https://doi.org/10.1007/s42773-025-00436-4>.

Supplementary material 1.

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## Author contributions

Conceptualization, Methodology [Pia Ramos], [Daniel Ashworth], [Mike Schmidt]; Investigation, Writing—original draft, Formal analysis [Pia Ramos]; Writing – review and editing [Daniel Ashworth], [Mike Schmidt], [Richeng Xuan]; Resources [Daniel Ashworth], [Mike Schmidt]; Project administration, Supervision, Funding acquisition [Daniel Ashworth].

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## Availability of data and materials

The authors declare that data supporting the findings of this study are available within the paper and in the Supporting Information. Should any raw data files be needed in another format they are available from the corresponding author upon request.

## Declarations

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### Competing interests

All authors declare that there are no competing interests to disclose.

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