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Research article

Biochar potential for long-term pharmaceutical remediation in flow-through tertiary wastewater systems

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ABSTRACT

Wastewater treatment plants are not designed to remove pharmaceuticals from wastewater and upon release pharmaceuticals end up in the environment. This study investigated the adsorption capacity of five different biochar materials under dynamic flow conditions utilising a packed bed column test. Tertiary wastewater effluent was used to evaluate the breakthrough of the biochar columns. Two conditions were tested during this experiment: (*i*) biochar remediation under high pharmaceutical loads, where wastewater was spiked with 16 pharmaceuticals; and (*ii*) biochar remediation under normal working conditions, where 42 non-spiked pharmaceuticals were evaluated. The concentrations of pharmaceuticals were measured over 45 weeks (635 bed volumes), and the adsorption capacity was assessed by plotting relative concentration against bed volumes. The results show a high adsorption efficiency (>99 %) for the sewage sludge and forest biomass biochar, emphasising the role of packing density, feedstock and physicochemical properties of the biochar. Furthermore, fitted breakthrough curves allowed for the assessment of the adsorption capacity of individual pharmaceuticals. The results demonstrate a strong correlation between the charge and molecular size of pharmaceuticals and their adsorption capacity. This study provides key insights into the remediation potential of biochar for the removal of pharmaceuticals in wastewater effluent.

1. Introduction

The presence of pharmaceuticals in wastewater effluent remains a significant challenge for wastewater treatment plants (WWTPs) (Angeles et al., 2020; Kosma et al., 2014). Despite improvements in treatment technologies, pharmaceuticals persist in treated effluent, raising concerns about their impact on human and environmental health (Patel et al., 2019). Conventional WWTPs are not specifically designed to remove these contaminants, leading to their continuous release into aquatic ecosystems (Angeles et al., 2020; Golovko et al., 2021; Kosma et al., 2014). As water scarcity and the need for resource recovery gain global attention, developing efficient strategies to eliminate or degrade pharmaceuticals from effluent are essential for sustainable water management.

The removal of pharmaceuticals is crucial to mitigate both human health and environmental risks. Pharmaceuticals present in treated effluent can contaminate drinking water sources, potentially leading to long-term health effects (Lind and Lind, 2020). The discharge of antibiotic residues into the environment is particularly concerning, as it contributes to antimicrobial resistance, a growing global health crisis (Zhuang et al., 2021). Additionally, the reuse of treated wastewater for irrigation may introduce these compounds into crops, raising concerns about dietary exposure and food safety (Wu et al., 2015). Antidepressants and hormones have been shown to alter the behaviour and reproduction of aquatic organisms, ultimately leading to population declines and biodiversity loss (Brodin et al., 2014). To address these concerns, advanced post-treatment of WWTP effluent is necessary to enhance pharmaceutical removal. This will allow the safe reuse of treated effluent for multiple applications; irrigation (Foghagen and Alriksson, 2024), industrial processes (Pintilie et al., 2016), groundwater recharge (Sui et al., 2015).

Among quaternary treatment technologies, biochar adsorption has emerged as a promising, sustainable method for removing organic micropollutants, including pharmaceuticals (Ihsanullah et al., 2022; Li

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et al., 2019; Thompson et al., 2016). Compared to conventional adsorbents such as granular activated carbon, biochar can exhibit comparable or even superior adsorption performance for hydrophobic and charge-dependent compounds, particularly in complex chemical mixtures (Huggins et al., 2016; Ihsanullah et al., 2022). As biochar is produced from renewable biomass or waste feedstocks, offering opportunities for carbon-negative wastewater treatment (Roberts et al., 2010; Thompson et al., 2016).

Despite its promise, biochar's effectiveness in removing pharmaceuticals under realistic flow conditions remains understudied (Amalina et al., 2022; Monisha et al., 2022). Most existing research evaluates biochar adsorption through batch experiments that focus on a limited selection of pharmaceuticals, which does not fully represent real-world wastewater treatment scenarios. To address these limitations, this study evaluates the long-term performance of five biochar materials for the removal of 42 pharmaceuticals from tertiary wastewater effluent under dynamic, flow-through, conditions that mimic real-world filtration. Additionally, targeted spiking of 16 pharmaceuticals, to simulate overflow events, which occur when WWTPs are hydraulically overloaded during storms or maintenance shutdowns and release untreated or partially treated effluent containing elevated contaminant loads (Kay et al., 2017; Petrie, 2021), were analysed to investigate the robustness of biochar. The experiment was conducted over a 45-week period, treating more than 600 bed volumes, utilising wastewater effluent to represent typical operating conditions. To the best of our knowledge, this is the first study to evaluate the long-term performance of a continuous-flow biochar filter treating tertiary wastewater effluent.

The five biochar materials used in this study were selected from a variety of locally available feedstocks in Sweden and the Nordic region, including sewage sludge, forest biomass, forestry residues, and agricultural seed waste. Overall, this study provides a comprehensive evaluation of biochar's capacity to remove pharmaceuticals from real tertiary effluent under dynamic conditions, highlighting long-term performance trends, feedstock-specific variability, and its potential role in circular wastewater treatment systems of the future.

2. Material and methods

2.1. Standards and chemicals

The detailed information about purchased standards, reagents, and chemicals can be found in text in Supplementary Information (SI). All analytical standards were of high analytical grade (>95 %). In total, 58 compounds were selected for analysis based on their frequent detection in municipal wastewater, environmental prevalence and spatial distribution in aquatic ecosystems, and production and consumption trends (Jiang et al., 2024; Malnes et al., 2022). Further details can be found in Table S1 of the SI.

2.2. Sample collection

Tertiary wastewater effluent was collected from the municipal WWTP in Uppsala, Sweden (200,000 Population Equivalent, PE), which utilises a combination of sand filtration, activated sludge and $FeCl_3$ flocculation for treatment (Uppsala Vatten, 2023). A total of 15 sampling events were conducted over a 10-month period, with approximately 75 L of wastewater collected every third week in 25 L high-density polyethylene containers. The first sample was collected on 25 January 2024 and the column experiment ran from 1 February 2024 to 16 December 2024. All samples were stored at 4 °C until use. Further information regarding the sampling can be found in Table S1 of the SI.

2.3. Biochar materials

Five biochar materials were used for the column experiment, as summarized in Table 1. The biochar materials were dried at 110 °C for 16 h and stored in sealed, waterproof containers prior to use. The dried biochar was used directly as sorbents in the column experiments without any additional preparation. Gravel, washed with hot water and MeOH, was used as a control material.

Table 1
The studied biochar materials and corresponding details. All columns are run in duplicate, and the average packing weight and standard deviation of each column is noted down.

Column	Weight (g)	Packing material	Production details
G	233 ± 4.2	Gravel for aquaria	Washed 3x with hot water and 3x with MeOH
SW	41 ± 4.1	Seed waste biochar	• Pyrolysis at 500 °C
PS	21 ± 1.8	Pine and spruce (mainly bark) biochar	Not disclosed
SS	104 ± 4.1	Sewage sludge biochar	• Pyrolysis at 600 °C • Time in reactors ~ 25 min
FB	76 ± 0.5	Forest biomass biochar	• Pyrolysis at 500–800 °C • Time in reactors ~3 h
SP	16 ± 0.6	Spruce biochar	• Pyrolysis at 650 °C • Time in reactors ~ 10 min

2.4. Column experiment

The column units, ran in duplicates, were constructed from PVC and had a total volume of 144.3 cm^3, with internal dimensions of 3.5 cm in diameter and a height of 15 cm for the sorbent compartment. Each column was packed by first adding gravel to the bottom, followed by a nylon mesh (50 μm) topped by a plastic filter, as depicted in Fig. 1. All columns were checked for leakage before use, and the biochar materials were preconditioned by hydrating them with Milli-Q water for 14 days.

Two experimental scenarios were evaluated: (*i*) treatment of unspiked tertiary wastewater effluent to assess the removal of 42 pharmaceuticals present under baseline conditions, and (*ii*) spiking of the effluent with 16 selected pharmaceuticals at a concentration of 100 μg/L to simulate a worst-case contamination scenario (Patel et al., 2019; Tran et al., 2018). The spiked compounds were selected to represent a broad range of physicochemical properties, including molecular mass, molecular formula, and charge state (see Table S2), ensuring a representative assessment of adsorption behaviour and breakthrough dynamics. This high-concentration spiking strategy was designed to simulate overflow conditions during heavy storm events or extreme hydraulic loads, when WWTPs may exceed their capacity and discharge untreated or partially treated effluent into receiving environments (Kay et al., 2017; Petrie, 2021). By combining typical and high pharmaceutical load scenarios, the experiment approach aimed to enhance the traceability of adsorption and breakthrough behaviours under environmentally relevant and stress-test conditions.

The columns were operated from February 1, 2024, to December 16, 2024, for a total run time of 45 weeks at a constant flow rate of 12 mL/h (~2 bed volumes per day) using an Ismatec™ IPC 12 peristaltic pump. This flow rate was selected to ensure relevant hydraulic conditions while maintaining a balance between realistic contaminant exposure times and experimental feasibility. Sampling was conducted for 30 min at 8:00, 14:00, and 20:00 during the first 7 days to obtain approximately 6 mL per column, followed by a reduced frequency of two daily samples at 8:00 and 20:00 for the next 7 days. From the third week, samples were collected every morning at 8:00, transitioning to weekday-only sampling after the fourth week. After 19 weeks, sampling frequency was reduced to twice per week, and after 30 weeks, the sampling duration was increased to 1 h to accommodate potential changes in breakthrough dynamics over time. Samples were collected in polypropylene (PP) tubes and stored at < -18 °C until further analysis.

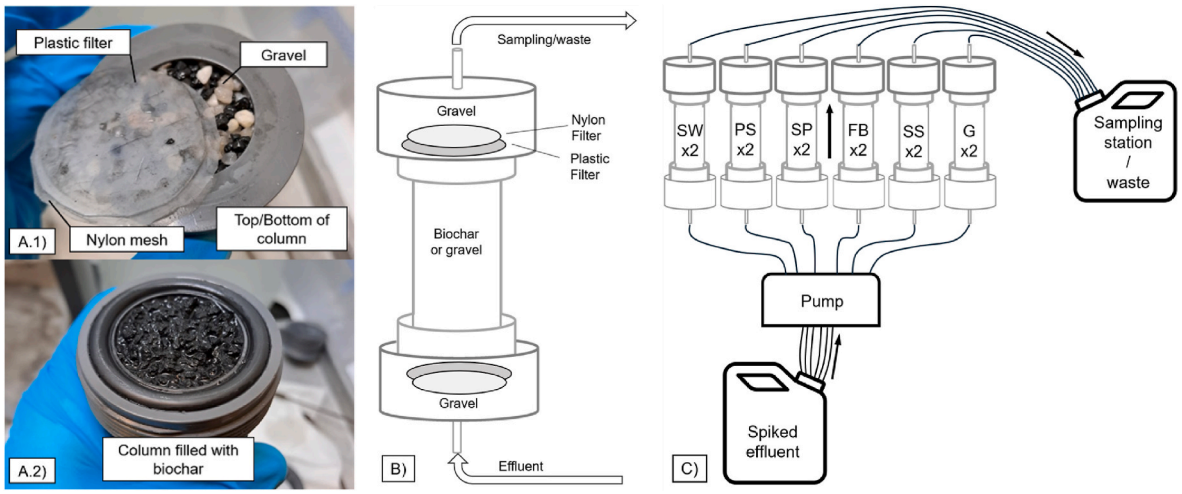

Fig. 1. Column experiment set up. A.1) The top of the column filled with gravel covered with the plastic filter and nylon mesh. This was placed on top of A.2) the column filled with biochar. B) A graphical depiction of the column setup. C) A graphical depiction of the full column setup with the flow going from the inlet vat to the pump, through the 12 columns and ultimately to the waste vat/sampling station.

2.5. Sample preparation and analysis

For the chemical analysis, 2.5 mL of the sample was filtered using a 0.45 µm regenerated cellulose syringe filter (Minisart® RC, Sartorius, Germany), and spiked with an internal standard mixture at final concentration of 50 ng/L. Laboratory blanks were prepared by filtering 10 mL of tap water and spiked with the internal standard mixture at 50 ng/L. Calibration curves were measured at the beginning and end of each sequence to check the instrument repeatability. Matrix effects were assessed for each compound and corrections for ion suppression or enhancement were accomplished using matrix-matched standards. Matrix matched standards were prepared by spiking sampled wastewater with both native and internal standard compounds at 1000 ng/L and 50 ng/L respectively. The detailed information about LOQ and matrix effect can be found in text in SI.

Samples were analysed using an Ultimate 3000 Ultra-High Performance Liquid Chromatography (UPLC) system (Thermo Scientific, Waltham, MA, USA) coupled to a triple quadrupole mass spectrometer (QUANTIVA, Thermo Scientific, Waltham, MA, USA). The system was configured for on-line solid-phase extraction (SPE) with automated extraction and tandem mass spectrometric (MS/MS) detection. Data acquisition was performed using Xcalibur software (Thermo Fisher Scientific, San Jose, CA, USA), and data processing and evaluation were conducted using TraceFinder™ 4.1 software (Thermo Fisher Scientific).

2.6. Biochar characterization

Different physico-chemical parameters of the biochar materials were measured. Elemental analysis of the biochar solids was performed in triplicate by a commercial lab. From there, aromaticity of the biochar materials was estimated by calculating the ratio between H and C. Additionally, B.E.T. surface area on this biochar was measured in another study (Celma et al., 2025). In brief, the microporous and the external surface area was measured by employing N_2 physisorption.

2.7. Data analysis

The relative concentration (C/C_0) of each pharmaceutical was calculated by comparing the pharmaceutical concentration in the column (C) to the average concentration measured in the control (gravel) columns (C_0). To evaluate the biochar adsorption, pharmaceutical breakthrough curves were modelled using the Yoon-Nelson model (Yoon and Nelson, 1984). The performance is comparable to other common models, such as the Thomas and Adams-Bohart models (Chu, 2020; Ndoun et al., 2023; Puga et al., 2022), mainly due to a high mathematical similarity (Croll et al., 2022) and were therefore excluded from this study. The Yoon-Nelson model is extensively used to determine the breakthrough time of carbon columns (Manjunath and Kumar, 2021) and can help to evaluate the retention capabilities of individual pharmaceuticals. Another issue with the Thomas and Adam-Bohart models is the requirement of a constant input concentration (C_0) (Chen et al., 2012). The numerical formula of the Yoon-Nelson model does not require this. Nevertheless, the Yoon-Nelson model is still based on a constant C_0. Therefore, only the Yoon-Nelson model was used to estimate the 20 % breakthrough point. The Yoon–Nelson equation is given as:

$$\frac{C}{C_0} = \frac{a}{1 + \exp\left(K_{YN}\tau - K_{YN}t\right)}$$

where K_{YN} is the Yoon-Nelson coefficient, τ the amount of bed volumes required to reach 50 % breakthrough, t the number of bed volumes and a the observed breakthrough limit. While the parameter a is typically set to 1, a scaled model, with boundaries set in this study between 0.4 and 2, can capture the complex variations, like the chromatographic effect, to a higher extend (Croll et al., 2022). This adjustment accounts for the complex nature of adsorption and desorption processes in biochar, as well as the potential influence of (bio)degradation and the formation of transformation products. The collected data exhibit substantial variability due to variations in wastewater effluent composition, temperature fluctuations, and both instrumental and experimental uncertainties. To reduce the influence of outliers, a robust regression approach utilising Tukey's bisquare (biweight) function was applied (Chen, 2020).

3. Results and discussion

3.1. Studied compounds in WWTP

For the column experiment, tertiary wastewater effluent was used as influent for the columns. Here the gravel/control columns were continuously monitored on the pharmaceutical concentrations. A summary of the pharmaceutical concentrations and their analytical assessments can be found in Table S4. Wastewater composition varies over different seasons and WWTPs, nevertheless these pharmaceuticals have been previously reported in both influent and effluent of WWTPs around the world (Beek et al., 2016; Golovko et al., 2021; Petrović et al., 2014; Verlicchi et al., 2012), highlighting the overall persistency of

pharmaceuticals globally. The maximum concentrations ranged from 5300 ng/L for hydrochlorothiazide (HCTZ) to 5.1 ng/L for diltiazem with several pharmaceuticals (like azithromycin, diazepam, miconazole) found below the LOQ. Pharmaceuticals detected at concentrations below the LOQ were excluded from further analysis during this study. A high variability is noticeable in the concentrations throughout this column experiment. These variations may be attributed towards seasonal variation in the wastewater effluent (Beek et al., 2016; Kibuye et al., 2019), biofilm formation, biodegradation processes (Li et al., 2019) and analytical uncertainties associated with low-concentration compounds.

To evaluate the background concentration of spiked compounds in wastewater, analysis of non-spiked tertiary wastewater was performed, with the results presented in Table S4. One of the spiked compounds, salicylic acid, could not be detected using the current method and was excluded from further analysis. This study did not focus on the seasonal variability of pharmaceuticals in non-spiked wastewater, as previous research has extensively documented such trends (Golovko et al., 2014). Nevertheless, throughout the experiment, the concentrations of the target compounds (spiked and non-spiked) were continuously monitored in the control column to assess the adsorption efficiency and effluence of the real-life fluctuations.

3.2. Characterization of the biochar materials

Table 2 summarises the measured physicochemical properties of the 5 different biochar materials. In terms of elemental composition, most biochar materials are showing similar C %, while only SS biochar containing a relatively low amount of C (29.9 %). However, this difference is not as pronounced when evaluating the H and N %, highlighting the fact that several other chemical elements should be present in SS composition to compensate for its remarkably low C%. This could potentially be due to the feedstock of SS being sewage sludge and, thus, having a more complex chemical mixture than usual plant-based feedstock materials. In contrast, there are no large differences in the amount of H per material, which results in an increased H/C ratio for SS biochar in comparison with the other materials. This higher ratio indicates a higher degree of polarity in the biochar composition which could yield more prevalent polar interaction with pharmaceuticals (Tong et al., 2019). In any case, similar values for the aromaticity can be found in other studies for SS (Lu et al., 2013) and plant based biochar materials (Chen et al., 2008; Weber and Quicker, 2018).

The B.E.T. surface area, as reported in Celma et al. (2025), show for both FB and SP biochar the largest surface area for both external and microporous indicating that such materials hold a larger space for molecular interaction with the pharmaceuticals. Contrarily, the surface area for SW and SS are significantly lower than those for FB and SP (up to 40 times lower), thus limiting their potential interaction with contaminants for their retention. On the other hand, no large differences are observed for the average adsorption pore size of the materials.

3.3. The adsorption capacity of different biochar materials

The global breakthrough curve was constructed by taking the average breakthrough of the duplicates, representing the median C/C_0 values for all pharmaceuticals at each sampling point. Additionally, the first and third quartiles were included to illustrate variability across compounds. The global breakthrough curves are presented in Fig. 2. The breakthrough curves show that the studied biochar materials have a large variability in the adsorption capacity of pharmaceuticals (Fig. 2) with some biochar materials, like SW, PS and SP, showing a high relative concentration of pharmaceuticals (such as oxazepam and carbamazepine) while other materials such as SS and FB maintain a high adsorption capacity along the tested bed volumes.

In this context, the breakthrough curves indicate the limited retention capacity in the SP biochar (Fig. 2E). Complete breakthrough ($C/C_0 = 1$) was observed after approximately 20 bed volumes, leading to the early termination of these columns. On the contrary, Fig. 2C and D demonstrate the high adsorption capacity of the SS, and the FB biochar materials, all of which maintained a >99 % ($C/C_0 > 0.01$) removal efficiency after 635 bed volumes. The variation in overall performance underscores the significant influence of feedstock type and the physicochemical properties of biochar on its sorption capacity for pharmaceutical removal (Ahmad et al., 2014).

One notable difference among these three biochar materials is their packing density in the columns, which varies due to differences in particle size. While this variation exists, we cannot conclusively determine its impact on adsorption performance, as no direct data was collected to assess its influence. In this experiment, the biochar materials were used in their original form without pre-processing to alter the particle size. Previous studies have suggested that smaller particle sizes, which inversely correlates with packing density, can enhance adsorption rates due to an increased active surface area (He et al., 2022; Raposo et al., 2009). While post-processing methods, such as particle size reduction, could potentially optimize adsorption performance (Sangani et al., 2020), these approaches introduce additional costs and labour, limiting their feasibility for large-scale applications. The impact of packing density on the adsorption performance was not determined as no direct data was obtained. Studies show that biochar composition changes overtime when submerged (Spokas et al., 2014), so future research could involve a non-invasive analysis technique like X-ray tomography combined with a tracer liquid as it could provide high-resolution data on pore structure, preferential flow channels, and spatial adsorption patterns over time.

Another potential factor influencing adsorption efficiency is the formation of preferential flow channels, which can occur at lower packing densities. This phenomenon reduces overall contact time between the wastewater and the biochar, potentially decreasing adsorption efficiency (Hill and P, 1952). However, without direct measurements, the extent of this effect remains uncertain.

In addition to physical structure, the chemical properties of biochar contribute to differences in adsorption capacity (Tong et al., 2019). The FB biochar exhibited the highest carbon content (C%) and a relatively high hydrogen-to-carbon ratio (H/C), indicative of a low oxygen-to-carbon ratio (O/C) and a high pyrolysis temperature. Such conditions typically yield biochar with more aromatic structures and greater resistance to pH fluctuations, with adsorption dominated by π-π

Table 2
The weight percentage of Carbon (C), Hydrogen (H) and Nitrogen (N), the molar Hydrogen/Carbon (H/C) ratio was measured by an external lab for this study. Additionally, both external and microporous B.E.T. surface area of the analysed biochar materials,[a] as measured in another study (Celma et al., 2025). SW: Seed Waste Biochar. PS: Pine and Spruce Biochar. SP: Spruce Biochar. FB: Forest Biomass Biochar. SS: Sewage Sludge Biochar.

Biochar	C%	H%	N%	H/C	B.E.T. surface area[a]		Average adsorption pore size[a] (nm)
					Microporous (m^2/g)	External (m^2/g)	
SW	71.7 ± 2.7	2.4 ± 0.02	3.2 ± 0.9	0.40	6.3	8.1	8.0
PS	76.9 ± 14.8	0.95 ± 0.2	0.3 ± 0.1	0.15	44.5	30.2	2.8
FB	88.8 ± 2.3	2.3 ± 0.07	0.07 ± 0.06	0.31	250.1	83.8	2.4
SS	29.9 ± 0.7	1.3 ± 0.05	1.9 ± 0.04	0.50	14.9	21.5	6.9
SP	84.2 ± 2.5	2.1 ± 0.02	0.04 ± 0.05	0.30	237.8	41.5	2.3

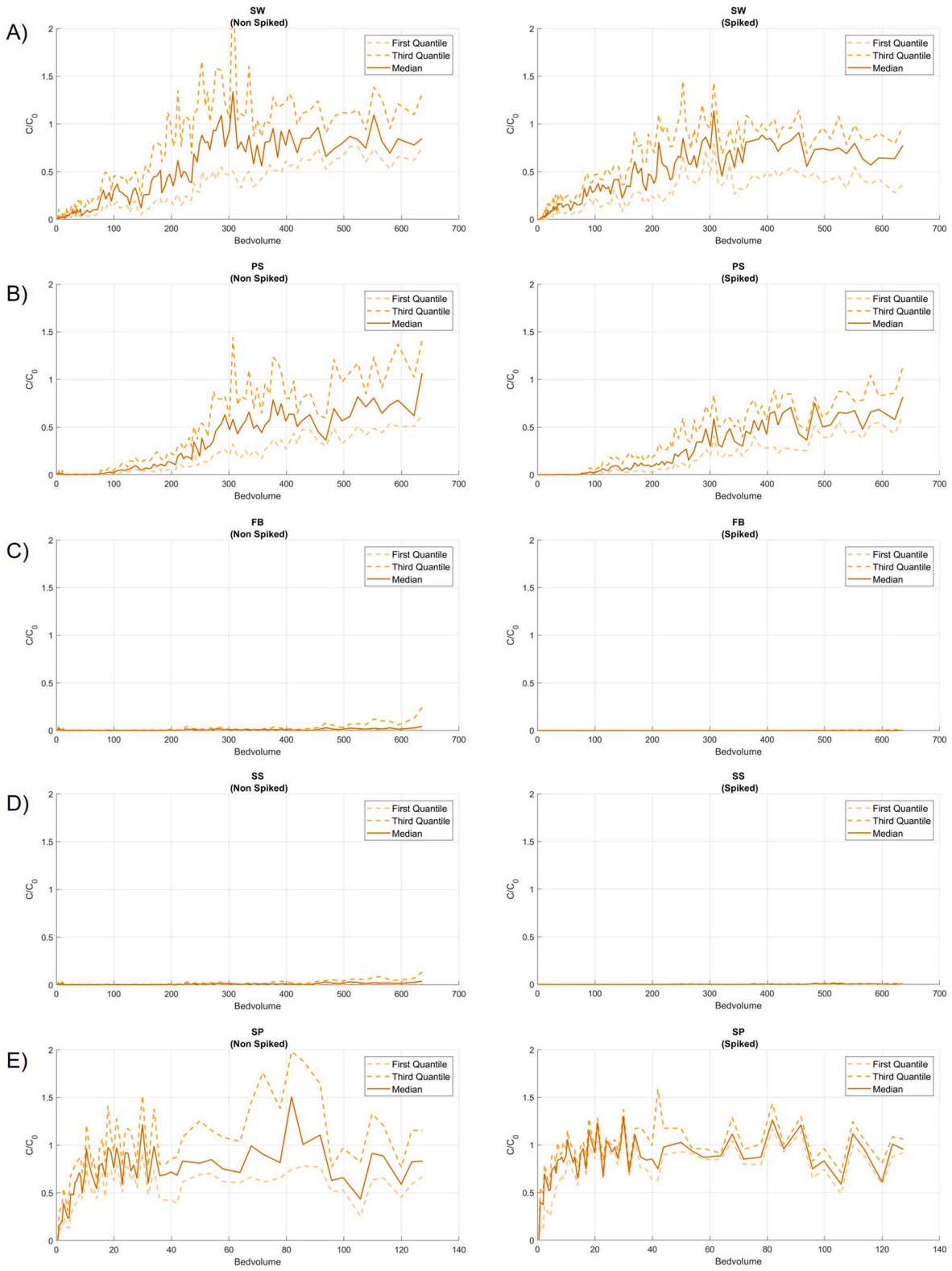

Fig. 2. The relative concentration (C/C_0) plotted against the bed volumes of wastewater effluent the columns have been exposed to. The median, the first and third quantile of the C/C_0 are depicted for spiked (left column) and non-spiked (right column) pharmaceuticals in the 5 different analysed biochar materials. SP was only measured until bed volumes 137 due to its high breakthrough rate. A) SW: Seed Waste Biochar. B) PS: Pine and Spruce Biochar. C) FB: Forest Biomass Biochar. D) SS: Sewage Sludge Biochar. E) SP: Spruce Biochar.

interactions and hydrophobic forces (Lian and Xing, 2017). These factors may explain the effective and robust adsorption observed in FB biochar throughout the study.

Metals and especially iron in biochar can provide an improved adsorption due to added electrostatic interaction (Nakarmi et al., 2022). In WWTP's Ferric Chloride is commonly used to treat wastewater. Sedimentation will end up in the sludge and upon pyrolysis Iron will be mixed with the SS biochar. Even though no elemental analysis was performed, studies have shown that biochar made from sewage sludge show a high Iron content (Lu et al., 2013), potentially improving the overall adsorption capacities of the biochar (Zhang et al., 2023).

Surface area and pore volume are known parameters affecting the adsorption of pharmaceuticals, with steric hindrance occurring when pore diameters are smaller than the molecular size, and increased desorption arising from excessively large pores. (Zhu et al., 2020). While sludge biochar materials have usually larger pores compared to wood-based biochar materials (Ahmad et al., 2014; Skjennum et al., 2024), SS biochar pore size is in line with those of the plant-based materials in this study. Thus, distinct impact of pore size on the studied remediation of pharmaceuticals by biochar sorption can be excluded. Contrarily, large differences were observed for the surface area of the materials as above discussed. In general, the larger the surface area the higher capacity to retain chemicals (Ndoun et al., 2023); however, SS biochar showed one of the largest retention capacities while having the second lowest surface area, putting stronger emphasis on the mixed chemical composition of the material being responsible for the elevated retention of pharmaceuticals. Unlike lignocellulosic feedstocks, sewage sludge is a more complex biomass (Ihsanullah et al., 2022), which upon pyrolysis creates a complex carbon matrix with a variety of functional groups. These support hydrogen bonding, electrostatic interactions and other complex binding interactions, on top of π–π interactions. Nevertheless, FB biochar showed the largest surface area among the studied materials, which could be responsible for the enhanced capacity to retain chemicals with almost no break-through after 635 BV.

3.4. Pharmaceutical adsorption effects

To better evaluate the long-term capacity of biochar as a remediation tool, the relative concentration data was modelled to estimate the throughput (bed volumes) at which the breakthrough is 20 % (C/C_0 = 0.2). In the column experiment, two scenarios were evaluated: (*i*) spiking the effluent with 16 pharmaceuticals (100 μg/L) to simulate burdened events and (*ii*) analysis of tertiary wastewater effluent for 42 target compounds.

3.4.1. Sorption of pharmaceuticals under burdened events

Fig. 3 presents breakthrough curves for oxazepam, and carbamazepine as representative examples of the modelling conducted for the spiked pharmaceuticals. The observed differences are attributed to the diverse physicochemical properties of the pharmaceuticals, which influence their adsorption capacity and interaction with the biochar (Zietzschmann et al., 2016).

The full set of fitted Yoon-Nelson parameters is summarized in Table S5 of the SI. Smaller compounds (molecular weights 160–300 Da), such as tramadol, atenolol, and nicotine, exhibit breakthrough behaviour comparable to that of oxazepam. In contrast, verapamil and clarithromycin (490.3 Da and 747.4 Da), the largest molecules spiked in the wastewater, showed the highest retention. This indicates that larger, hydrophobic molecules are more effectively retained, potentially due to diffusion-controlled adsorption within pores (Xiao and Pignatello, 2015).

Caffeine and nicotine exhibited distinct behaviour compared to the other spiked pharmaceuticals, showing a decrease in relative concentration after approximately 300 bed volumes (Fig. S1). This decline may be attributed to external factors, such as enhanced biodegradation or photodegradation during summer (Patel et al., 2019) or background contamination (Liachenko et al., 2015), as both compounds are widely consumed stimulants in Sweden.

In general, the breakthrough curves exhibit fluctuations in behaviour, with the final breakthrough values stabilizing either above or below one. These variations may be attributed to sudden peaks and drops in wastewater effluent concentrations. For example, losartan, was only spiked until bed volume 216 to simulate the effects of a sudden drop in influent concentration (Fig. S2). Despite this reduction, the C/C_0 remained elevated during the longevity of this experiment, indicating continued leakage from the biochar. This persistent breakthrough suggests even a substantial reduction in the influent concentration of a pharmaceutical may not lead to immediate reduction in effluent

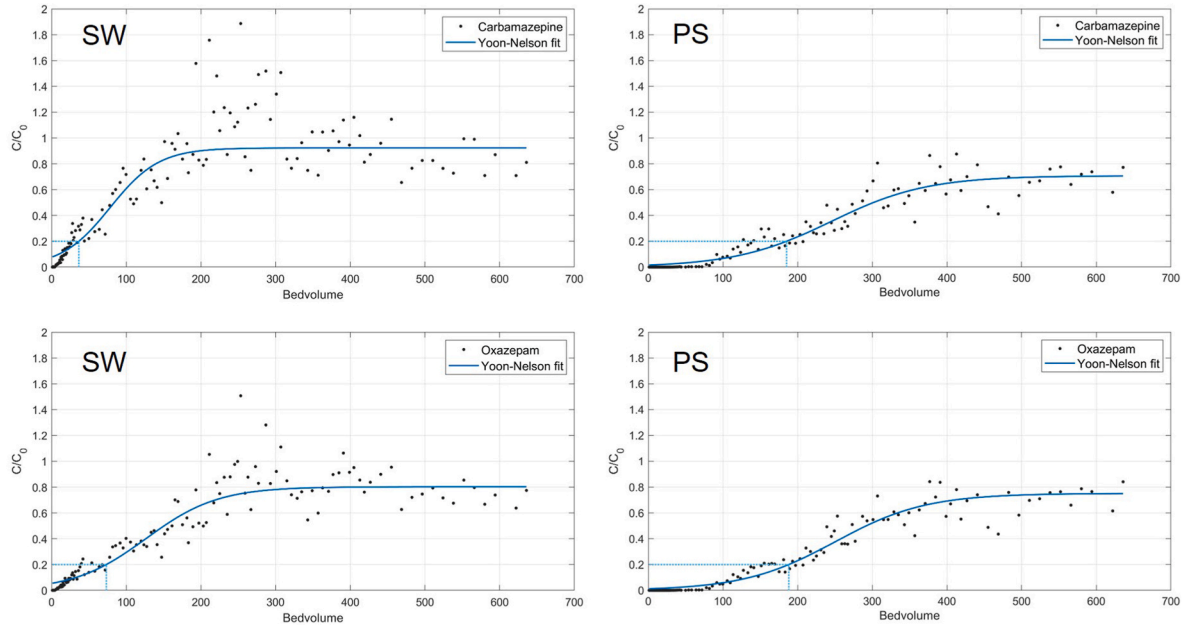

Fig. 3. Breakthrough curves of carbamazepine (top) and oxazepam (bottom) for the Seed Waste Biochar (SW) and the Pine and Spruce Biochar (PS). Fitting was done using the Yoon-Nelson model and the breakthrough 20 % (C/C_0 = 0.2) was estimated, see the light blue lines in the graph.

concentrations, especially when other organic micropollutants continue to compete for limited adsorption sites.

In addition to concentration dynamics, biological processes may also play a crucial role in shaping the breakthrough behaviour. As wastewater flows through the columns, microbial communities start to attach to the biochar and walls of the column, forming a biofilm (He et al., 2020). Biofilm is known to actively support the biodegradation of pharmaceuticals and other micropollutants, decreasing the concentration output (Korotta-Gamage and Sathasivan, 2017). Biologically active carbon is known to even have a so-called 'self-cleaning' ability (Smolin et al., 2020). This lowers not only the output but also cleans up the biochar for new adsorption sites, stabilizing the relative concentration ratio below one. Nevertheless, the biofilm can become too large and start to block the water flow. Pressure will build up in the system, causing the biofilm structure to burst. As a result, contaminants and bacteria can flow out causing a spike in contaminant concentrations (Simpson, 2008). Several precautions were implemented in this study to minimize biofilm formation and associated operational issues. The influent wastewater reservoir was replaced bimonthly to limit microbial growth and reduce the risk of biofilm development within the storage container. In addition, all tubing was replaced three times over the 45-week experimental period to prevent clogging. Throughout the study, no significant pressure buildup or visible channel blockage was observed, indicating effective maintenance of flow conditions.

3.4.2. *Biochar sorption capacity under normal WWTP working conditions*

Of the 42 target compounds analysed in untreated wastewater effluent, breakthrough curves were modelled for 22 pharmaceuticals to determine the bed volume at which 20 % breakthrough occurred. Most pharmaceuticals exhibited a significant decrease in concentration during the summer period, falling below the LOQ, likely due to reduced pharmaceutical intake and improved wastewater treatment (Golovko et al., 2014). Other pharmaceuticals were detected only at very low concentrations, making their analysis challenging, particularly given the complex nature of the wastewater matrix. Therefore, only a selection of 22 non-spiked pharmaceuticals could be modelled and the summary of the results can be found in Table S5.

There are several therapeutical groups that are extensively found in wastewater effluent. The adsorption of antibiotics (erythromycin, clindamycin and the spiked clarithromycin) onto biochar is exemplified by erythromycin in Fig. 4. Antibiotics often have a high molecular volume compared to other pharmaceuticals Previous studies have shown that size is an important factor in the retention onto carbon columns (Sörengård et al., 2020), where a smaller molecular volume shows a faster initial adsorption, but also a faster desorption (Xiao and Pignatello, 2015).

Anti-depressants (citalopram, desvenlafaxine, fluoxetine, furosemide, venlafaxine and the spiked amitriptyline), are smaller (<350 Da) cationic species and show in general great retention onto the biochar columns, Fig. 4. Biochar is known to have a high density of negatively charged species on its surface (Oh and Seo, 2016), and this could explain their improved retention. Some anti-depressants, like citalopram and fluoxetine, were detected in the raw wastewater effluent during the start of this study; however, the concentration dropped below the LOQ when spring approached and were excluded from this study.

Another therapeutical group are the cardiovascular drugs (bisoprolol, metoprolol and the spiked propranolol). This group is widely used and found in wastewater effluent (Zhang et al., 2020). Fig. 5 shows the relative concentration of metoprolol, which belongs to the class of β-blockers. Other β-blockers, like bisoprolol, show a high retention onto the biochar. This group consist of long chained, cationic molecules, which promotes not only diffusion into the micropores, but also electrostatic interaction (Ndoun et al., 2023). Additionally, these pharmaceuticals contain a benzene-ring, which could enhance π- π interactions with the biochar (Ndoun et al., 2023).

The cardiovascular sartan-family (irbesartan and valsartan) showed large variation throughout this experiment. Valsartan is known to be more strongly affected by biodegradation compared to other sartans (Bayer et al., 2014), which could explain the individual variations. Commonly, this family has negatively charged groups at higher pH levels (>7.4), which could prevent adsorption onto the negatively charged biochar surface. Irbesartan has a pKa of 7.4 and small fluctuations in the pH could swing the charge from negative to neutral, leading to fluctuations in the adsorption rate. Influent wastewater pH ranged from approximately 8.5–9.0 during the initial phase of the experiment and gradually decreased, stabilizing between 7.5 and 8.5 in the later stages. While these minor pH fluctuations may have contributed to day-to-day variability in the adsorption of certain ionizable

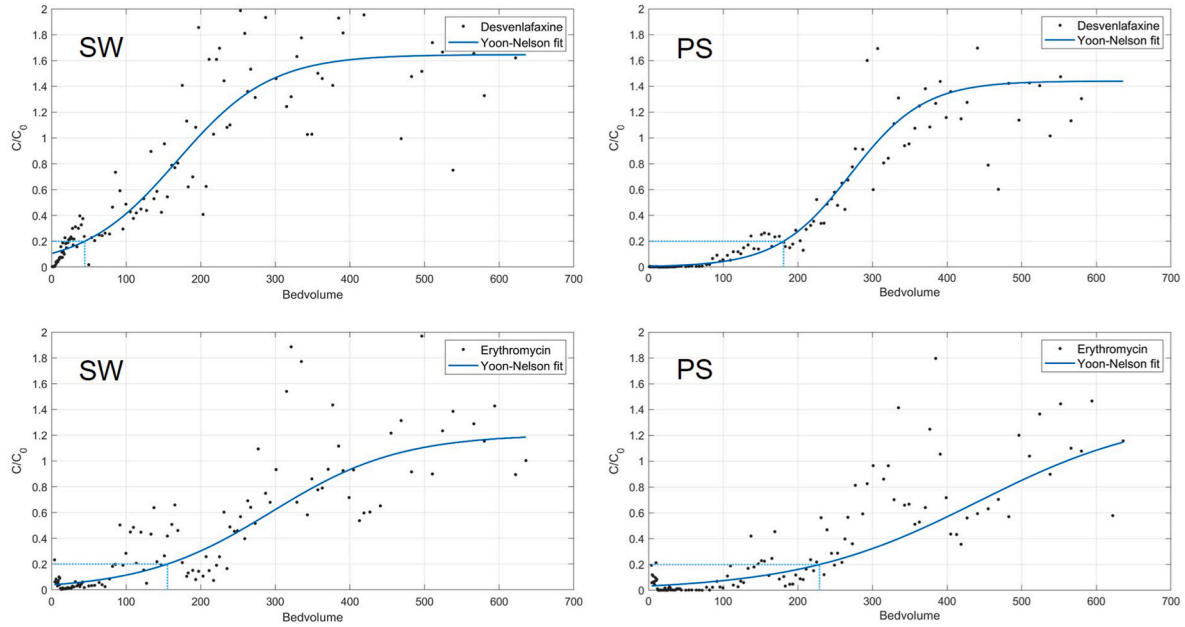

Fig. 4. Breakthrough curves of desvenlafaxine (top) and erythromycin (bottom) for the Seed Waste Biochar (SW) and the Pine and Spruce Biochar (PS). Fitting was done using the Yoon-Nelson model and the breakthrough 20 % ($C/C_0 = 0.2$) was determined, see the light blue lines in the graph.

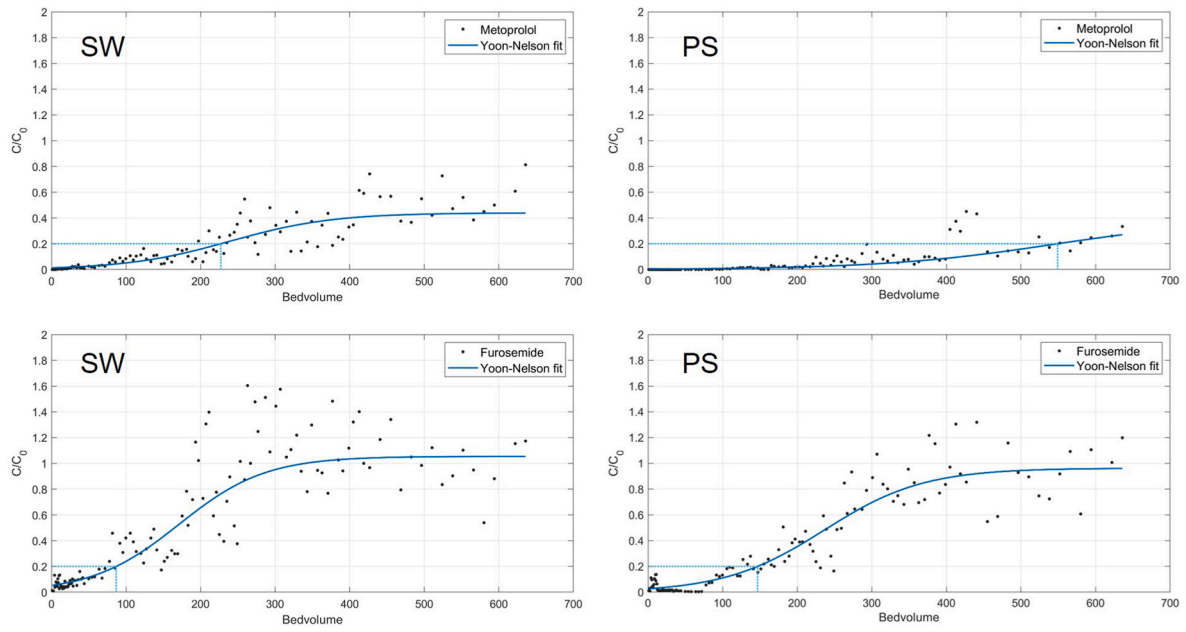

Fig. 5. Breakthrough curves of metoprolol (top) and furosemide (bottom) for the Seed Waste Biochar (SW) and the Pine and Spruce Biochar (PS). Fitting was done using the Yoon-Nelson model and the breakthrough 20 % ($C/C_0 = 0.2$) was determined, see the light blue lines in the graph.

pharmaceuticals, they are unlikely to account for the more substantial differences in adsorption performance observed across the various biochar types.

Other cardiovascular pharmaceuticals, like the antilipemic drugs (atorvastatin and the spiked bezafibrate) are generally anionic, where the spiked bezafibrate shows limited retention onto the biochar. Other antilipemic pharmaceuticals, like atorvastatin, show a larger retention presumably due to its size.

The studied antihistamines (cetirizine and the spiked fexofenadine) show an above average adsorption onto biochar. This could be attributed to amphoteric nature of the studied antihistamines, where they contain a positive and a negative charge, increasing the retention by binding not only on negative sites, but also potential positive sites.

Diuretics (furosemide and HCTZ) are a class of pharmaceuticals that contain generally a lot of functionalised groups, including sulfonyl and amine groups. Which could lower the retention on the biochar materials with a higher carbon content, due the decreased polarity (Ahmad et al., 2012). This therapeutical group also has a relatively low molecular weight leading to a relative low retention compared to previously mention pharmaceuticals. In Fig. 5 the relative concentration of furosemide is shown as an example.

The adsorption behaviour of the studied pharmaceuticals seems to be strongly influenced by their molecular weight and charge, as shown in Fig. 6. Pharmaceuticals with a negative charge and low molecular weight generally exhibit a lower retention compared to larger, neutral or positively charged, molecules. However, biochar adsorption is a complex process involving multiple interactions based on different physicochemical properties, including π-π interactions and hydrogen bonding. Nonetheless, biochar performance and adsorption capacity can be effectively monitored using a select group of high-abundance, low molecular weight compounds (<350 Da) such as carbamazepine and furosemide.

3.5. Environmental application

The results of this study have shown to be beneficial for wastewater treatment and the mitigation of pharmaceutical contaminants in aquatic ecosystems. The persistence of pharmaceuticals in wastewater effluents remains a critical environmental concern, as these bioactive compounds can disrupt ecological processes and pose risks to aquatic biota. Conventional WWTP technologies often demonstrate limited efficiency in removing these contaminants, necessitating the development of supplementary and more sustainable remediation strategies.

This study demonstrates that biochar-based adsorption is a viable approach for the removal of pharmaceutical contaminants, offering a potential alternative to existing treatment technologies. Among the five biochar materials evaluated, two exhibited particularly high adsorption capacities, with sludge-derived biochar demonstrating the most promising performance. Given the widespread availability of sewage sludge (European Commission, 2023), its valorisation into biochar could present a future opportunity to establish a circular economy within WWTPs. The utilization of sludge-derived biochar as an adsorptive medium could

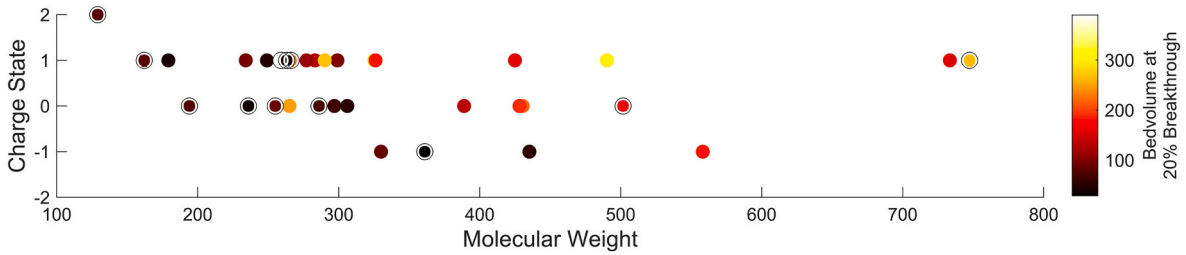

Fig. 6. Molecular weight plotted against the Charge state of all studied pharmaceuticals at pH 8. Spiked pharmaceuticals are shown with a black circle. The pharmaceuticals are coloured with the 20 % breakthrough of the Pine and Spruce (PS) biochar.

enhance WWTP efficiency while simultaneously mitigating the environmental impact associated with sludge disposal, however more research on life cycle assessment and large-scale pilot studies is required.

A critical factor in the long-term application of biochar filtration is the acceptable level of leakage from the columns. As part of the new European wastewater directive, urban wastewater treatment plants bigger than 100,000 PE are required to implement quaternary treatment, aiming for a minimum of 80 % removal efficiency of certain target OMPs (European Parliament, 2024). Our findings indicate that biochar can effectively retain over 80 % of the pharmaceuticals over an extended period, highlighting its potential as an alternative solution for quaternary treatment. Additionally, the European Parliament has established a list of target pharmaceuticals to monitor removal efficiencies (European Parliament, 2024). FB and SS biochar demonstrated high retention for some of the target compounds, making biochar a promising material for quaternary wastewater treatment.

Nevertheless, the adsorption capacity of biochar is finite, and over time, desorption and leakage of pharmaceuticals will occur as sorption sites become saturated. One of the European target compounds, carbamazepine, exhibited among the lowest retention on biochar in this study, making it a useful indicator for evaluating long-term performance of biochar. To ensure the feasibility of large-scale biochar applications, it is essential to estimate the longevity of the biochar that effectively polishes WWTP effluent. Here the adsorption capacity observed in this study could provide a solid basis for a larger pilot application.

4. Conclusions

This study evaluated the breakthrough behaviour of biochar columns for the removal of pharmaceuticals from wastewater effluent. Temporal and spatial variability in influent wastewater composition is an inherent challenge in real-world treatment systems. However, this variability represents a strength of the present study, which was specifically designed to assess the performance of various biochar materials under dynamic, long-term, and operationally realistic conditions. Among the tested materials, sewage sludge (SS) and forest biomass (FB) biochar exhibited the highest removal efficiencies, achieving up to 99 % removal over 635 bed volumes. Breakthrough curve analysis revealed substantial variability in adsorption performance between biochar, influenced by material-specific properties such as packing density, pore size, and aromaticity, factors related to the feedstock type and pyrolysis conditions. The Yoon-Nelson model captured differences in how individual pharmaceuticals adsorbed to the biochar and enabled estimation of the 20 % breakthrough point, aligning with the European wastewater directive for quaternary treatment. Spiking experiments demonstrated the robustness of biochar under high contaminant loads, underscoring its potential for treating burdened wastewater scenarios. The findings highlight the critical role of the physicochemical properties of pharmaceuticals, particularly charge and molecular weight, in determining adsorption efficiency. This study reinforces the use of biochar as an alternative quaternary treatment option and provides a solid foundation for future work aimed at optimizing biochar longevity, refining breakthrough models, and scaling up for long-term application.

CRediT authorship contribution statement

Bent Speksnijder: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Alberto Celma:** Writing – review & editing, Data curation, Conceptualization. **Maximilian Tyka:** Methodology, Formal analysis. **Prithvi Simha:** Writing – review & editing, Data curation, Conceptualization. **Oksana Golovko:** Writing – review & editing, Supervision, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jenvman.2025.127389.

Data availability

Data will be made available on request.

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