

Activated carbon and biochar prepared from date palm fiber as adsorbents of phosphorus from wastewater

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ARTICLE INFO

Keywords:

Wastewater treatment
Phosphorus removal
Activated biochar
Biochar
Date palm fiber

ABSTRACT

Activated carbon derived from agriculture waste has recently gained widespread use due to its high efficiency, especially in wastewater treatment applications. The article aims to enhance the wastewater treatment process by evaluating advanced treatment within secondary treatment stages, using activated biochar technology and biochar. It also investigates the removal efficiency when sand is added to both activated biochar and biochar, and finally compares the results with an existing wastewater treatment plant.

First, biochar and activated biochar were prepared from a bio-based precursor (date palm fiber). Then, four types of adsorbent media were prepared: the first consists of activated biochar, the second of activated biochar with added sand (75 % biochar: 25 % sand by volume), the third of biochar with sand added (75 % biochar: 25 % sand by volume), and the fourth of biochar alone. All media were tested as adsorbents for total phosphorus from actual raw wastewater under identical hydraulic conditions. All media demonstrated good potential for total phosphorus removal, with activated biochar showing the highest removal efficiency (81–91 %), followed by activated biochar with sand (68–86 %), biochar with sand (71–85 %), and finally biochar alone (59–83 %). Furthermore, all types of media were resilient to simulated hydraulic and phosphorus overloading events.

1. Introduction

Water scarcity is a serious issue affecting communities worldwide due to drought and increasing water demand, making the conservation of every drop essential. Wastewater accounts for approximately 80–90 % of total water use, so effective treatment is necessary to produce a clear effluent that can be reused in agriculture, for instance, or at least safely discharged into surface water without harming the environment [1]. Domestic wastewater consistently contains organics, solids, microorganisms, heavy metals, and nutrients, all of which pose significant risks to both human health and the environment. Among the primary environmental concerns, with substantial and immediate impacts, is the destruction of marine habitats [2]. One of the harms of discharging wastewater into waterways is eutrophication—the enrichment of water bodies with nutrients, leading to a decline in water quality due to excessive plant growth and the resulting disruption to ecological

balance. Phosphorus is typically the limiting nutrient in freshwater lakes, reservoirs, and rivers, and human-induced phosphorus inputs accelerate the eutrophication process. In addition to the above, global supplies of phosphate rock are steadily declining, and phosphorus plays a critical role in global food security. Ensuring food security and meeting legal and regulatory requirements for phosphorus recovery from wastewater are becoming increasingly urgent [3]. Human excreta, household garbage, and the usage of phosphorus-rich washing products (including sodium phosphate and poly-sodium phosphate) are the primary sources of phosphorus in residential sewage. The current discharge limits for total phosphorus (TP) are approximately 1–2 mg/L. In traditional WWTPs, between 10 % and 30 % of the P may be removed by biological processes and solids settling [4]. So, various methods have been developed to eliminate excess phosphorus from wastewater, with the aim of mitigating its detrimental impact on the ecosystem. Various physical, chemical and biological techniques have been extensively

Abbreviations: BOD, biochemical oxygen demand; CAB, chemically activated biochar; COD, chemical oxygen demand; EC, electric conductivity; TP, total phosphorus; TWW, treated wastewater; RWW, raw wastewater.

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<https://doi.org/10.1016/j.dwt.2024.100925>

Received 22 September 2024; Received in revised form 18 November 2024; Accepted 27 November 2024

Available online 30 November 2024

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employed to eliminate total phosphorus (TP) from wastewater. Chemical techniques include use of metal salts [5] advanced oxidation processes (AOPs) such as chlorination [6], ozonation [7], Fenton reaction [8], ion exchange [9], chemical precipitation [10], and adsorption [11].

Biological techniques include enhanced biological uptake [12], Enhanced Biological Phosphorus Removal (EBPR) which polymerizes phosphate into polyphosphate by utilizing polyphosphate-accumulating organisms have been widely used and are considered mature for phosphorus removal [13].

Among the several methods for eliminating contaminants, adsorption with solid materials—referred to as adsorbents—is an easy, practical, and efficient procedure. Mineral, organic, or biological materials might be adsorbent matter. Over the past thirty years, a variety of methods utilizing unconventional adsorbents have been investigated in an effort to develop more affordable and efficient adsorbents for the removal of contaminants at detectable levels [14]. Some of these trials were to use different agricultural wastes to reduce phosphorus concentrations [15] [16]. Ghezzehei [17] revealed that phosphate concentration in flushed dairy manure can be reduced by 19–65 % with activated biochar made from agricultural waste. Some other trials using biochar with sand showed 75–83 % removal efficiency from secondary treated wastewater [18]. Compared to commercial activated biochar, low-cost adsorbents derived from various sources generally exhibit limited adsorption capability for removing contaminants. Consequently, the pursuit of cost-effective materials for use as adsorbents and precursors for activated biochar production remains ongoing [19]. The aims to enhance the wastewater treatment process by evaluating advanced treatment within secondary treatment stages, using activated biochar technology and biochar. It also investigates the removal efficiency when sand is added to both activated biochar and biochar, and finally compares the results with an existing wastewater treatment plant. The phrase "biochar" is a relatively new scientific term. "A carbon (C)-rich product when biomass such as wood, manure, or leaves is heated in a closed container with little or unavailable air," according to Lehmann and Joseph [20]. Activated biochar is a refined version of biochar that undergoes chemical or physical treatments to enhance its adsorption properties, making it particularly suitable for environmental applications such as water and wastewater treatment. These modifications significantly improve its ability to remove organic pollutants, heavy metals, nutrients, and other contaminants from water. Biochar and chemically activated biochar (CAB) have attracted considerable interest in their roles in environmental remediation and agriculture. Biochar, a carbon-rich material formed through pyrolysis in oxygen-limited conditions, is particularly effective at adsorbing contaminants due to its unique characteristics, including a large surface area, well-developed pore structure, high porosity, ion exchange capacity, and significant carbon content [21]. Many studies investigated biochar preparation. These studies found that changes in the structure and physicochemical characteristics of biochar are closely associated with the temperature at which pyrolysis occurs. They also assured that While biochar produced at 400 °C contains volatile and readily labile components, biochar produced at 600 °C develops a highly resistant nature. They also confirmed that biochar produced at 400 °C contains volatile and labile components, whereas biochar produced at 600 °C exhibits a more resistant structure [22–24].

Biochar activation can be performed using various techniques, each offering distinct advantages and tailored applications. These techniques include physical activation, chemical activation, and a hybrid approach that combines both methods. Chemical activation involves the use of activating agents such as KOH, H₃PO₄, ZnCl₂, CuCl₂, and sub/supercritical CO₂ to create activated biochar from a variety of precursors. These precursors include materials like polyacrylonitrile, sludge, rice straw, petroleum coke, orange peel, tamarind pulp, macadam [25–31]. Chemical activation is generally more cost-effective than physical activation due to its lower activation temperature, reduced processing time, and higher carbon efficiency. Furthermore, chemically activated

biochar more easily develops a porous structure. Among chemical activators, phosphoric acid requires a lower activation temperature compared to potassium hydroxide and is less harmful to the environment and human health than zinc chloride [32].

The pilot plant of this study was installed at the Qaha wastewater treatment plant in Egypt and operated for 9 weeks from June 5, 2022, to August 4, 2022. The pilot plant was working continuously, 24 h a day, over a nine-week period.

2. Materials and methods

2.1. Biofilter media preparation

Fine sand was sourced from a nearby construction supply store in Markaz Kafr Shukur. To remove larger particles and contaminants, the sand was sieved through a 2 mm mesh. It was then washed with tap water to eliminate dissolved organic matter and fine particles.

2.2. Agriculture waste preparation

Solid waste made from date palm fiber was collected from a date palm tree in Benha, Alqaluobia. For 24 h at 105 °C, the solid waste was dried in the oven. Then the biochar and activated biochar were prepared.

2.2.1. Biochar

In the lab, biochar was produced through a gradual pyrolysis process. Date palm fiber was placed in crucibles, and the crucibles were sealed with aluminum foil to limit oxygen exposure. A small needle hole was made in the foil to allow evolved gases to escape. The crucibles were then placed in a muffle furnace, where the temperature gradually increased from room temperature to 550 °C at a rate of 17 °C per minute. Once the final pyrolysis temperature was reached, it was maintained for an additional 1.5 h. Afterward, the biochar was allowed to cool within the furnace. In total, two batches of biochar were prepared.

2.2.2. Chemically activated biochar (CAB)

The first step involved releasing the cellulose and trace components from the date palm fiber. The fiber was immersed in a 5 % sulfuric acid (H₂SO₄) solution for 24 h. The floating dust on the surface of the acidic solution was decanted. After that, the samples were washed several times with distilled water to remove any remaining acid and then dried. Next, 100 mL of freshly prepared 30 % phosphoric acid (H₃PO₄) solution was mixed with approximately 50 g of the date palm fiber particles. This mixture was then placed in the furnace for activation, where the temperature was maintained at 600 °C for two hours. After cooling to room temperature, the sample was washed repeatedly with distilled water until the pH reached between 5 and 7.

2.3. The characterization of activated biochar and biochar

The activated biochar's fundamental physical and chemical properties were studied in the lab. By combining 1 g of activated biochar with 20 mL of distilled water, the pH was ascertained, and the pH reading was taken using the Xplorer GLX ps-2002 instrument. [18] EC was measured using the same samples used for the pH analysis. By adding activated biochar to a 10 mL graduated cylinder and tapping it until it achieved constant volume, the bulk density was calculated.

2.4. Treatment preparation

Four column reactors were prepared. The first column reactor was filled only with CAB. The second column reactor is filled with CAB mixed with sand (CAB comprised 25 % of the total column volume) as per previous studies mentioned in literature review (Bradley et al., 2015). The third column reactor is filled only with biochar. The fourth

column reactor is filled with biochar mixed with sand (biochar comprised 25 % of the total column volume). The pilot was prepared as shown in Fig. 1.

2.5. Packing of media

The described setup shown in Fig. 2 for the biofilter column involved using a 70-mm internal-diameter glass column. A drainage layer of washed crushed stone was placed at the bottom to ensure proper flow. To avoid mixing of the media layers, two layers of plastic mesh were placed between each. The biofilter media, consisting of either a sand/activated biochar mixture or pure activated biochar, was packed into the column to a height of 150 mm. To prevent the activated biochar from floating, another two layers of plastic mesh were added, followed by a 20-mm layer of crushed stones. This arrangement ensured uniform distribution of wastewater across the filter surface during the experiment.

2.6. Loading of the columns

The pilot study was conducted at the Qaha wastewater treatment plant in Qaha city, located in Al-Qalyubia, Egypt. This plant uses an oxidation ditch for biological treatment and processes municipal wastewater from the city's residents, hospitals, schools, and nearby villages. Raw wastewater (RWW) from the plant's grit removal system was used as the influent for the pilot study. {{Fig. 3}}.

The RWW was stored in a feeding tank, which supplied the columns at the designated hydraulic loading rate (HLR) through valves. Each column received an average daily wastewater volume of 7000 mL, corresponding to a daily HLR of $1.875 \text{ m}^3/\text{m}^2$. The daily total phosphorus (TP) loading rate was $7.5 \text{ g P}/\text{m}^2$. The system operated from Sunday to Thursday and rested on Friday and Saturday. Every week, three samples of both influent and effluent were collected for analysis.

Between weeks 4 and 6, the system underwent flooding simulations by increasing the hydraulic load by about 30 %. The new daily loading rate was 9100 mL, equivalent to a daily HLR of $2.37 \text{ m}^3/\text{m}^2$, to simulate high hydraulic loading during heavy rainfall events. Samples were collected and analyzed similarly to the first three weeks. In the final three weeks of the study, the system returned to its initial loading of 7000 mL of wastewater per day to observe its behavior under normal conditions. This approach aimed to assess the biofilter system's performance under varying loading conditions and its capacity for treating wastewater.

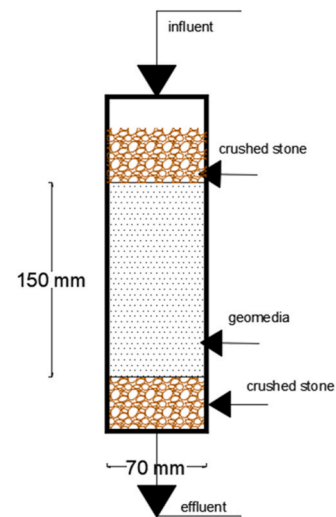


Fig. 2. Schematic diagram of the column design.

2.7. Measuring of total phosphorus

TP in an aqueous sample can be determined, by turning phosphorus compounds into orthophosphate during sample digestion, so samples were first digested, then total phosphorus was measured using UV-Vis spectrophotometry. [33].

3. Results and discussion

The concentration of TP in the raw wastewater was 4–5.5 mg P/L. This experiment showed a very good possible removal of TP. Chemically activated biochar with no sand showed the best removal efficiency. But all treatments showed resistance to flooding events and stability of the system after the flooding ended. The subsequent sections provide a more comprehensive analysis of the results.

3.1. Media characterization

Several removal mechanisms, such as physical adsorption through pores, electrostatic attraction through binding sites, chemical sorption through a chemical reaction with binding sites, and surface precipitation through salts, are thought to be involved in the biochar adsorption process for nitrate and phosphate [34]. So, there was necessary to

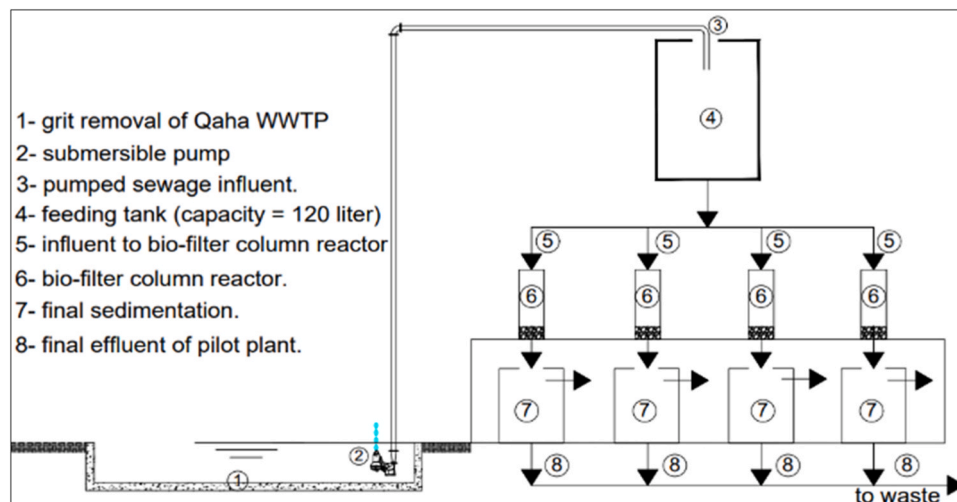


Fig. 1. schematic diagram of the pilot.

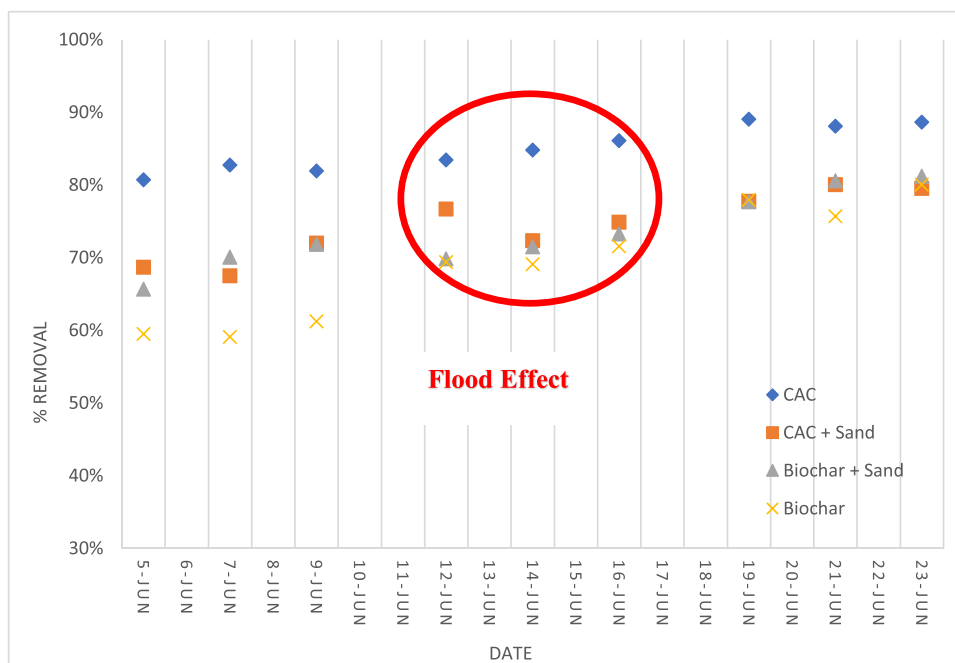


Fig. 3. TP removal efficiency of the influent and effluent from each treatment.

investigate some properties of the two materials.

Some of the fundamental physical characteristics of the activated biochar and biochar used in the experiment are listed in Table 1. From this table, we can deduce that:

1. $Yield(\%) = \frac{m_a}{m_b} * 100\%$ where:

m_a is the weight of sample after preparation.

m_b is the weight of sample before preparation.

The yield for both biochar and CAB was relatively low this due to Dehydration and the temperature-induced fractionation of biomass into components including cellulose, hemicellulose, and lignin [35] More specifically, condensation polymerization is responsible for the decline in biochar output at rising temperatures [36].

2. The ash content was measured after burning the samples to a constant weight in a muffle furnace at 750 °C for 6 h. As the temperature of pyrolysis rises, condensation polymerization takes center stage. At low temperatures, crop residues' macromolecular components could not completely break down, and only a small portion of their weak chemical connections were destroyed. Crop waste loses moisture and hydrate water before 250 °C, and hemicellulose breaks down between 200 and 300 °C. In this instance, crop waste might only produce trace amounts of gas and tar that are made up of numerous tiny molecules. Subsequently, the breakdown of cellulose takes place between 300 and 380 degrees Celsius, while the breakdown of lignin happens between 200 and 500 degrees Celsius. As the temperature rises, the molecular bonds of the high polymer components break and more volatile stuff is produced [36].
3. Specific surface area for CAB is larger than biochar as Important process variables that affect the surface area are the chemical impregnation ratio and the ultimate activation temperature. When

the activation temperature is raised, the activation process increases the development of existing pores and generates new ones, increasing the BET surface areas [37]. According to the previous experimental findings, using activating agent (H_3PO_4) created new pores when the right impregnation ratio was used. The porosity produced by acid and water washing is thought to be caused by the gaps left by phosphoric acid, as indicated by the increase in surface area and pore volume with the impregnation ratio [38].

4. Based on the outcomes of batch studies [39] and [40] revealed that the pH range of 6–10 was ideal for the removal of phosphate from aqueous solutions using activated biochar. The procedure employed in this experiment is within the ideal pH range, as shown in Table 2.

3.2. Effluent pH

The TWW (influent) had a pH between 7.6 and 8.2, The pH values of the effluent from CAB ranged from 7.0 to 9.0, CAB + sand ranged from 7.1 to 8.8 and biochar + sand 7.4 to 9.8 and biochar ranged from 7.5 to 9.8 indicating considerable alkalinity.

The results revealed that the average pH value of the biochar is greater than that of CAB. This may be associated with the chemical activation of CAB. And the decrease in the pH value when adding sand. This observation is in agreement with [41,42] who noted that after filtering with biochar, the pH of the wastewater from a dairy lagoon increased. The higher pH of the filter media is probably the cause of the higher pH of the effluent. Also, Because of the production of $Ca_3(PO_4)_2$ in the higher pH environment, the inclusion of the alkaline biochar raised the pH of the solution, which may have aided phosphate precipitation [43].

3.3. Effect of different media on removal of TP

Table 3 illustrates the total phosphorus concentrations in mg/l of the influent and effluent from each treatment. Table 4 illustrates removal efficiencies and depicts the removal efficiencies of each medium under different loading circumstances. CAB was the best medium for TP removal (81–91 %) then CAB with sand (68- 86 %) then biochar with sand (71–85 %) then biochar (59–83 %).

These results for phosphorus removal by biofiltration are in line with

Table 1

Activated biochar and biochar physical and chemical properties.

property	biochar	CAB
Yield (%)	19.34 ± 0.81	16.88 ± 0.53
Ash content (%)	8.62 ± 1.03	9.06 ± 0.76
Specific Surface Area (m ² /g)	253 ± 9.5	500 ± 24
pH	6.5 ± 0.24	6.9 ± 0.45

Table 2
pH of the influent and effluent from each treatment.

Date	RWW	biochar		CAB		Biochar + Sand		CAB + Sand	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD
9-Jun	7.4	7.8	0.25	7.9	0.39	7.8	0.12	7.5	0.19
16-Jun	7.8	8.3	0.29	7.6	0.29	8.1	0.25	8.5	0.19
23-Jun	7.2	8.4	0.15	7.8	0.14	7.6	0.10	8.5	0.33
30-Jun	7.5	8.3	0.25	8.0	0.20	8.3	0.47	7.8	0.25
7-Jul	7.6	8.6	0.30	7.8	0.33	8.5	0.40	7.6	0.25
14-Jul	8.3	9.5	0.33	8.6	0.36	9.4	0.44	8.4	0.27
21-Jul	7.4	7.9	0.27	7.3	0.27	7.7	0.24	8.0	0.18
28-Jul	7.2	8.2	0.29	7.5	0.32	8.2	0.39	7.3	0.24
4-Aug	7.8	8.2	0.12	7.9	0.20	7.8	0.21	8.3	0.41

SD, Standard Deviation.

Table 3
TP concentrations in mg/l of the influent and effluent from each treatment.

	Raw	CAB	CAB + Sand	Biochar + sand	Biochar	WWTP eff.
5-Jun	3.5643	0.687	1.116	1.223	1.444	3.303
7-Jun	3.7052	0.639	1.203	1.109	1.515	3.492
9-Jun	3.6612	0.661	1.025	1.032	1.420	3.220
12-Jun	3.268	0.541	0.762	0.986	1.001	2.698
14-Jun	3.1001	0.471	0.857	0.884	0.958	2.713
16-Jun	3.005	0.417	0.754	0.803	0.854	2.894
19-Jun	3.4002	0.372	0.755	0.758	0.750	3.321
21-Jun	3.3702	0.401	0.672	0.655	0.819	3.251
23-Jun	3.412	0.388	0.698	0.642	0.680	3.298
26-Jun	4.7783	0.555	0.841	0.904	0.994	4.432
28-Jun	4.5227	0.626	0.890	0.920	0.983	4.227
30-Jun	4.544	0.566	0.821	0.899	0.900	4.365
3-Jul	4.424	0.544	0.790	0.820	0.921	4.202
5-Jul	4.331	0.599	0.740	0.888	0.896	4.113
7-Jul	4.303	0.494	0.677	0.745	0.824	4.089
10-Jul	3.951	0.395	0.597	0.621	0.674	3.202
12-Jul	3.982	0.382	0.578	0.594	0.645	3.113
14-Jul	4.13	0.420	0.568	0.610	0.650	3.089
17-Jul	4.33	0.519	0.659	0.870	0.833	4.000
19-Jul	4.745	0.481	0.674	0.796	0.923	4.440
21-Jul	4.56	0.431	0.692	0.744	0.824	4.230
24-Jul	5.02	0.552	0.768	0.905	0.994	4.770
26-Jul	4.894	0.470	0.698	0.784	0.758	4.560
28-Jul	4.751	0.481	0.713	0.775	0.792	4.431
31-Jul	3.914	0.416	0.601	0.691	0.682	3.740
2-Aug	4.092	0.350	0.636	0.744	0.715	3.823
4-Aug	4.159	0.370	0.639	0.623	0.726	3.854

the reported values in the literature. Bradley and Hanandeh [39,41] did, however, confirm that although biochar in the sand media reduced the manure leachate’s TP removal efficiency. Biochar, which typically has a low anion exchange capacity, was present in the biochar [44]. However, alternative mechanisms play a major role in the elimination of phosphorus, as it is not as reliant on ion exchange [45,46]. However, biochar typically has negatively charged surfaces [44]. This reduces their

Table 4
TP removal efficiency of the influent and effluent from each treatment.

	CAB	CAB + Sand	Biochar + Sand	Biochar
5-Jun	81 %	69 %	66 %	59 %
7-Jun	83 %	68 %	70 %	59 %
9-Jun	82 %	72 %	72 %	61 %
12-Jun	83 %	77 %	70 %	69 %
14-Jun	85 %	72 %	71 %	69 %
16-Jun	86 %	75 %	73 %	72 %
19-Jun	89 %	78 %	78 %	78 %
21-Jun	88 %	80 %	81 %	76 %
23-Jun	89 %	80 %	81 %	80 %
26-Jun	88 %	82 %	81 %	79 %
28-Jun	86 %	80 %	80 %	78 %
30-Jun	88 %	82 %	80 %	80 %
3-Jul	88 %	82 %	81 %	79 %
5-Jul	86 %	83 %	79 %	79 %
7-Jul	89 %	84 %	83 %	81 %
10-Jul	90 %	85 %	84 %	83 %
12-Jul	90 %	85 %	85 %	84 %
14-Jul	90 %	86 %	85 %	84 %
17-Jul	88 %	85 %	80 %	81 %
19-Jul	90 %	86 %	83 %	81 %
21-Jul	91 %	85 %	84 %	82 %
24-Jul	89 %	85 %	82 %	80 %
26-Jul	90 %	86 %	84 %	85 %
28-Jul	90 %	85 %	84 %	83 %
31-Jul	89 %	85 %	82 %	83 %
2-Aug	91 %	84 %	82 %	83 %
4-Aug	91 %	85 %	85 %	83 %

affinity to negatively charged ions like phosphate due to repulsion and makes them more appealing to positively charged cations [46]. Because of competition for adsorption sites from cations in wastewater as well as other anions that are more ion-exchangeable, like NO^{-3} , Cl^{-} , and SO^{-4} , phosphorus removal may be lessened as a result. This explains why the biochar media removes less phosphorus than sand. Furthermore, the Ca^{+2} and Mg^{+2} content of the biochar affects its ability to adsorb phosphorus, according to [47]. Because of the production of $\text{Ca}_3(\text{PO}_4)_2$ in the higher pH environment, the addition of alkaline biochar raised the pH of the solution, which may have facilitated phosphate precipitation [43]. Also, the results make it clear that the biomass size had an impact on the physical properties of the resultant activated biochar. This suggests that other properties of the activated biochar, such as surface functional groups, surface charge, and specific surface area, may have also been impacted, which would have increased the activated biochar’s ability to absorb phosphorus than other media.

3.4. Flooding effect on the TP removal

From week 4 to week 6, there were significant flooding occurrences that affected the treatments. To determine the system’s stability under flooding conditions, the system’s performance following the flooding incident was assessed. Lucas and Greenway [48] found that bioretention

mesocosm performance improved after significant rain events. Ali El Hanandeh¹, Ammar, and Mamoun reported that after the flushing operations, the removal efficiency remained consistently high [18].

In this study, it was observed that the effectiveness of TP removal wasn't affected. Ali El Hanandeh, Ammar A. Albalasmeh, and Mamoun Gharaibeh reported that after the flushing operations, the removal efficiency remained consistently high [18].

4. Conclusion

Two types of adsorbents were prepared from date palm fiber; the first was made by physical pyrolysis, and the second was activated chemically. The produced activated biochar and biochar were used as filter media, and by mixing each type with sand, we got two more types of filter media to test their efficiencies on TP removal. CAB was the best medium for TP removal (81–91%) then CAB with sand (68–86%) then biochar with sand (71–85%) then biochar (59–83%). Furthermore, all types of media were resistant to simulated hydraulic and phosphorus overloading events. The findings reported carry significant ramifications for agricultural waste usage and wastewater treatment methods that rely on the use of activated biochar applications. However, despite this, further future studies should be conducted to ensure the stability of the biochar prepared in nutrient removal. Additionally, the experiment should be applied to a larger scale and over a longer period to confirm that no clogging occurs in the medium.

CRedit authorship contribution statement

Sameh Mansour: Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. M.E. Basiouny: Writing – review & editing, Validation, Supervision. O.A. Abosiada: Supervision, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

Data availability

Data will be made available on request.

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