

Bamboo-derived biochar as an efficient adsorbent for oxytetracycline removal from water

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ABSTRACT

The increasing presence of oxytetracycline (OTC) in water sources due to its extensive use in aquaculture and livestock rearing necessitates effective removal techniques. We evaluated the adsorption performance of two forms of bamboo-derived biochar, one produced through CO₂ activation (B1) and the other produced through steam activation (B2), in both batch and continuous systems. The results showed that bamboo-derived biochar exhibited low ash content and high fixed carbon, with the biochar produced by steam activation (B2) demonstrating better porosity, as evidenced by lower bulk density and bee-hive structures observed in the SEM images. Batch experiments showed that B2 exhibited a higher maximum adsorption capacity (34.3 mg/g) compared to B1 (21.5 mg/g) under optimized conditions. Continuous column experiments revealed that B2 achieved superior performance, with a breakthrough time of 50 h and a maximum adsorption capacity of 100 mg/g, significantly higher than B1's breakthrough time of 20 h and capacity of 44 mg/g. Under re-used conditions, B2 out-performed B1 with Ce/C₀ stabilizing at ~0.1, compared to ~0.4 for B1, highlighting the enhanced efficiency of B2 over repeated cycles. However, after regeneration using distilled water, both materials exhibited a significant decline in adsorption performance, with Ce/C₀ values exceeding 0.8, indicating the ineffectiveness of the regeneration method. These results emphasize the structural advantages of B2, contributing to its superior adsorption performance in both batch and continuous systems. Despite these strengths, the limited reusability of both materials underscores the need for optimized regeneration protocols.

1. Introduction

Bamboo, a plant belonging to the Poaceae family, is classified under the subfamily Bambusoideae. Renowned for its rapid growth, bamboo is one of the fastest-growing plants in the world, capable of extending several centimeters per day depending on the species and environmental conditions (McClure, 2013). According to the Food and Agriculture Organization (FAO)'s Global Forest Resources Assessment 2020 (FRA), bamboo covers approximately 35 million hectares of land across Africa, Asia, and the Americas (Global Forest Resources Assessment, 2020, 2020). Vietnam's warm and humid climate provides ideal conditions for bamboo growth (Permaculture Plants, 2014). The country ranks fourth globally in bamboo area, with 194 species across 26 genera identified by

scientists (Bamboo Resources of Vietnam Internet). Bamboo culms exhibit a strong fibrous structure, high durability, and excellent bending resistance, making them widely used in construction and furniture manufacturing (Yap et al., 2017). Additionally, bamboo serves as a renewable energy source for gasification processes (Nguyen, 2024).

Over the past decade, significant research has been conducted on bamboo and its applications. Bamboo culms, when carbonized at temperatures between 600°C and 1000°C, can be transformed into activated carbon with high efficiency for CO₂ adsorption (Huang et al., 2014). When activated with CO₂ at 800°C, bamboo-derived materials show great potential as electrodes for Electric Double-Layer Capacitors (EDLC), a type of energy storage device (Khuong et al., 2021). In addition, bamboo treated with ZnCl₂ and pyrolyzed at 500°C has been effectively used to remove Cd(II) and Cr(VI) ions from wastewater (Chu

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Nomenclature		A	Ash Content
B1	Biochar 1	NOR	Norfloxacin
B2	Biochar 2	BD	Bulk Density
BOD	Biological Oxygen Demand	OTC.HCl	Oxytetracycline Hydrochloride
COD	Chemical Oxygen Demand	PFO	Pseudo-First-Order
EU	European Union	R-P	Redlich–Peterson
FAO	Food and Agriculture Organization	TGA	Thermogravimetric Analyzer
FDA	Food and Drug Administration	FC	Fixed Carbon Content
FRA	Forest Resources Assessment	SEM	Scanning Electron Microscope
ISO	International Organization for Standardization	MOX	Moxifloxacin
MRL	Maximum Residue Limit	PSO	Pseudo-Second-Order
EDLC	Electric Double-Layer Capacitors	M	Moisture Content
		V	Volatile Matter

et al., 2019). Pyrolysis at 400°C has also been investigated for enhancing water filtration systems, significantly improving the removal of impurities and bacteria from water sources (García-Ávila et al., 2023). Moreover, bamboo has been employed as a cost-effective adsorbent for wastewater treatment (Narzary et al., 2024).

Antibiotics, particularly oxytetracycline, are widely used in livestock and aquaculture to prevent and treat diseases as well as to promote growth. According to the OIE (2021) report, the global use of antibiotics in livestock is significant, with approximately 3000–4500 tons of oxytetracycline utilized annually (Éloit, 2023). In aquaculture, oxytetracycline accounts for about 60 % of the total antibiotics used in fish and shrimp farming (Lulijwa et al., 2020). Oxytetracycline (OCT) residues in the environment can contribute to antibiotic resistance development and disrupt microbial communities. Additionally, exposure to these residues can alter the human microbiome, leading to adverse effects such as allergic reactions, chronic toxicity, and digestive system dysfunctions (Le et al., 2025).

In Vietnam studies by the residues of oxytetracycline and other antibiotics have detected in food products such as fish and shrimp at levels ranging from 12 to 200 µg/kg (Binh et al., 2018; Luu et al., 2021). Additionally, oxytetracycline residues have been found in wastewater (2.1 µg/L), surface water (0.5–1.8 µg/L), and soil (0.01–0.5 mg/kg), exceeding the safety thresholds established by various international standards. Specifically, the European Union (EU) has set a maximum residue limit (MRL) of 100 µg/kg for oxytetracycline in seafood (European Commission, 2010; Commission Regulation (EU) No), while Japan’s limit is 50 µg/kg (Ministry of Health, Labour and Welfare, 2012; Annual Health). In the United States, the FDA permits a higher MRL of 200 µg/kg for seafood products (US FDA, 2018; eCFR).

Currently, there are no specific international standards regulating antibiotic residues in livestock wastewater. However, in China, studies have proposed permissible oxytetracycline concentrations in wastewater ranging from 1 to 2 µg/L (Wei et al., 2011). In Vietnam, the National Technical Regulation QCVN 62-MT:2016/BTNMT governs livestock wastewater management, focusing primarily on physical and chemical parameters such as BOD, COD, total nitrogen, and total phosphorus, as well as microbiological indicators. Nevertheless, this regulation does not specifically address antibiotic residues.

Cheng Li et al. (2024) investigated a novel bamboo-based magnetic biochar composite for the efficient removal of the antibiotic norfloxacin (NOR) from water systems. The composite demonstrated a maximum NOR adsorption capacity of 458.43 mg/g, attributed to its large surface area of 1253.66 m²/g achieved through KOH activation. Another study (Ouyang et al., 2024) revealed that bamboo-derived biochar, produced via pyrolysis at 700 °C, features a high pore volume and average pore size. It exhibited excellent adsorption efficiency for quinolone antibiotics, including moxifloxacin, ciprofloxacin, and ofloxacin, with maximum capacities of 135.56 mg/g, 151.31 mg/g, and 116.40 mg/g, respectively. Bamboo biochar, synthesized via pyrolysis, effectively

removes tetracycline from aqueous solutions, achieving a maximum adsorption capacity of 333.33 mg/g (Chen et al., 2024). The adsorption process is influenced by pH, with optimal performance at pH 6, and follows a pseudo-second-order kinetic model, indicating chemisorption. These results highlight bamboo biochar as a cost-effective and sustainable solution for antibiotic contamination in water. The rising demand for oxytetracycline in Vietnam’s aquaculture sector highlights an urgent need for effective methods to eliminate this antibiotic from aquatic environments. In response, this study explores the use of locally sourced bamboo biochar to remove oxytetracycline, evaluating its performance in both static and dynamic systems to assess its real-world potential.

2. Material and methods

2.1. Material preparation and analysis

The study utilized *Bambusa blumeana* (thorny bamboo), a widely cultivated bamboo species in rural Vietnam. The bamboo was processed following the ISO 18135:2017 standards, cut into segments measuring 7–10 cm in length and 0.5–1 cm in thickness. Oxytetracycline.HCl (OTC.HCl), with a purity of 95 %, was obtained from Marklin and used directly without further purification. The raw bamboo and activated samples underwent various analyses to characterize their characteristics. Bulk density (BD) was measured using the ASTM E873 standard. Moisture content (M) was determined following ASTM D4442, volatile matter (V) was analyzed based on ASTM D3175, ash content (A) was measured using ASTM D3174, and fixed carbon content (FC) was calculated by difference. Ultimate analysis was conducted to estimate carbon (C), hydrogen (H), nitrogen (N) and Oxygen (O, calculated by difference) contents using a CHN analyzer (MT-5, Yanaco). Additionally, the morphology of the activated samples was examined using a Scanning Electron Microscope (JEM5310, JEOL Ltd.).

2.2. Physical activation process

Physical activation experiments were conducted in a laboratory using a Macro Thermogravimetric Analyzer (Macro-TGA). The system features a ceramic tube reactor measuring 110 × 7.5 cm (length × diameter), housed within a heating unit. The reactor is equipped with three independent heating zones (T₁, T₂, T₃) to ensure uniform temperature distribution. Gas flows into the system were regulated by separate flow meters.

During the experiments, the reactor was preheated to 900°C. Bamboo samples were placed on a sample holder, raised to the reaction position, and exposed to an activation atmosphere of either 100 % CO₂ or 100 % H₂O, supplied from specialized gas or liquid reservoirs. CO₂ flow was measured using a Brooks SLA5800 Series Revision mass flow meter with an accuracy of ±0.1 %. Water flow was controlled by a Brooks 5882/D1A1A3A001 liquid mass flow meter with an accuracy of

$\pm 1\%$ of the full scale. The activation process was carried out under two distinct conditions: At 900°C with a CO_2 flow rate of 5 L/min for 25 minutes, producing activated carbon labeled as B1; At 900°C with a vapor flow rate of 150 g/h for 25 minutes, producing activated carbon labeled as B2. After activation, the carbon samples were immediately immersed in distilled water to prevent oxidation, ensuring material quality and stability. Fig. 1

2.3. OTC removal experiment

The adsorption experiments were conducted using bamboo derived biochar in both batch and continuous systems to evaluate its performance under various operational conditions. For batch experiments, 20 mL OTC solutions (OTC.HCl in distilled water) with concentrations ranging from 50 to 400 ppm were mixed with 0.1 g bamboo derived biochar in 100 mL Duran flasks, shaken at 150 rpm for 2 hours. Adsorption tests also examined the effects of contact time (5 – 180 minutes), pH (4 , 6 , 8 , and 10). Each setup used 20 mL of 100 ppm OTC solution and 0.1 g of charcoal. Filtration through 20 – $25\text{ }\mu\text{m}$ filter paper followed each experiment to prepare samples for analysis. Adsorption isotherms were modeled using Langmuir, Freundlich, Temkin, and Redlich–Peterson equations to characterize adsorption behavior (Table 1). The equilibrium adsorption capacity in milligrams/gram (mg/g) is determined by the following equation:

$$q_e = \frac{(C_0 - C_e) \cdot V}{W} \quad (1)$$

Where C_0 and C_e represent the initial and equilibrium concentrations of the adsorbate, V is the solution volume in liters (L), and W is the weight of the material used in grams (g).

In this study, the pseudo–first order model, the pseudo–second order model, the Elovich model and the intra–particle diffusion model were employed to investigate the adsorption kinetic (Table 2).

Table 1

Adsorption isotherm models (Ayawei and Ebelegi, 2017).

Isotherm models	Equation	Parameters
Langmuir	$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \quad (2)$	K_L : Langmuir constant related to adsorption capacity (L/mg). $q_{\max}$: Maximum adsorption capacity (mg/g). R_L : Dimensionless separation factor.
	$R_L = \frac{1}{1 + C_0 K_L} \quad (3)$	
Freundlich	$\ln q_e = \ln K_f + \frac{1}{n_f} \ln C_e \quad (4)$	K_f : adsorption capacity (L/mg). n_f : Freundlich isotherm constant
Temkin	$q_e = \frac{RT}{B_T} (\ln K_T + \ln C_e) \quad (5)$	B_T : the Temkin constant (kJ/mol). K_T : Temkin isotherm constant (L/g), T is the absolute temperature (K). R is the gas constant ($8.314 \cdot 10^{-3} \text{ kJ/mol.K}$).
Redlich–Peterson	$\ln \frac{C_e}{q_e} = \beta \ln C_e - \ln A \quad (6)$	A : Redlich–Peterson isotherm constant (L/g). β : Redlich–Peterson parameter.

The continuous adsorption experiments utilized a unique column setup to evaluate the performance of activated biochars B1 and B2. Each column consisted of a 60 mL plastic syringe packed with 10 g of biochar, designed to minimize material loss during operation. To ensure a controlled flow, a #14 peristaltic pump tube was employed, operating at a flow rate of 1 L/hour with a steady speed of 60 rpm throughout the experiments. 4 – 5 mL of each sample solution was collected hourly. Each

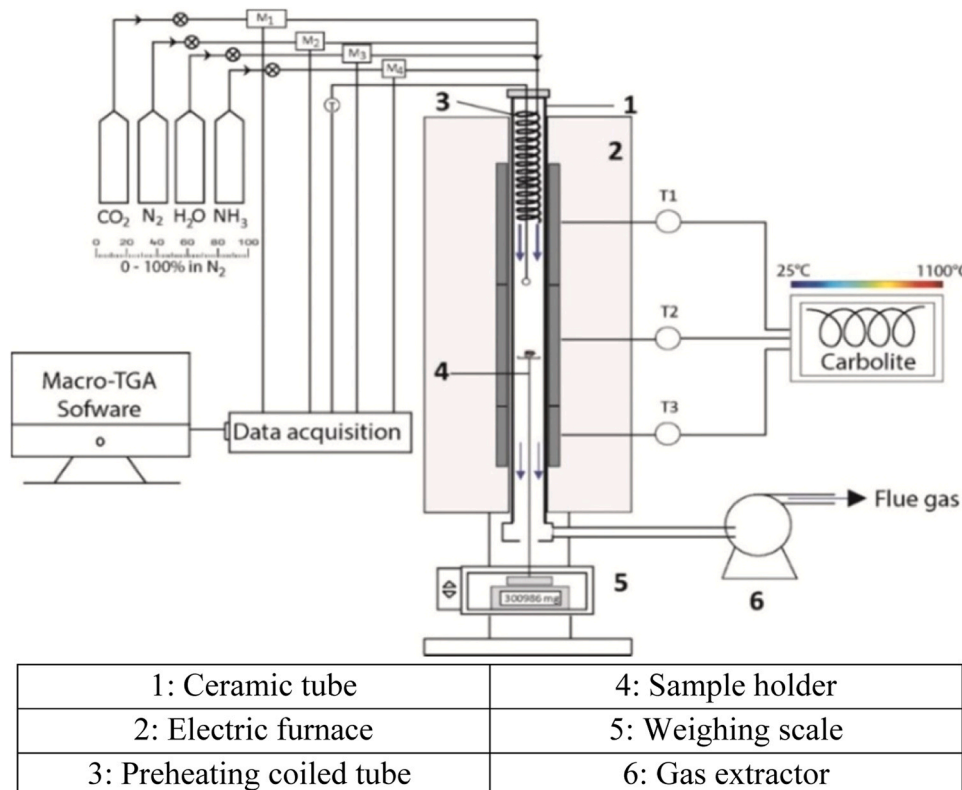


Fig. 1. Macro Thermo-Gravimetric Analyzer Gasification System used for physical activation.

Table 2

Kinetic models and equations (Tran et al., 2024).

Kinetic models	Equation	Parameters
Pseudo-first order	$q_t = q_e(1 - \exp(-k_1 t))$ (7)	k_1 : pseudo-first order kinetic model constant (L/min). q_t : amount of adsorbate adsorbed per mass of adsorbent at time t (mg/g).
Pseudo-second order	$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t}$ (8)	k_2 : pseudo-second order kinetic model constant (g/mg.min).
Elovich	$q_t = \frac{1}{\beta}(1 + \alpha\beta t)$ (9)	α : initial adsorption rate (mg/g.min). β : desorption constant (g/mg).
Intra-particle diffusion	$q_t = k_p t^{1/2} + C$ (10)	k_p : intra-particle diffusion rate constant (mg/g.min ^{0.5}). C : constant related to the boundary layer thickness.

sample was filtered through a fine filter to remove residual particulates, ensuring accuracy during subsequent UV-Vis analysis. OTC concentrations before and after adsorption were determined using a UV-Vis spectrophotometer (Shimadzu UV-1800) following Lambert-Beer law for precise measurement.

To calculate the maximum adsorption capacity ($q_{\max}$) of the biochars (B1 and B2) in the continuous system, the following formula can be used:

$$q_{\max} = \frac{F \cdot C_0 \cdot t_b}{m} \quad (11)$$

Where:

- F : Flow rate (L/h)
- C_0 : Initial concentration of OTC (mg/L)
- t_b : Breakthrough time (h), defined as the time until the effluent concentration (C_e) reaches a threshold (e.g., $C_e/C_0=0.9$).
- m : Mass of the biochar (g)

3. Results and discussion

3.1. Bamboo feedstock characteristics

Table 1 presents the comparative physicochemical properties of raw bamboo and its activated carbon, focusing on moisture content, bulk density, proximate analysis, and ultimate analysis.

The moisture content of activated carbons is lower than raw bamboo, which is expected due to the thermal activation process that removes water and other volatile compounds. Lower moisture content enhances the adsorption efficiency of activated carbon (Abd et al., 2021). Since H₂O molecules have a higher polarity and stronger affinity for many adsorbents, it can occupy the active sites or condense in micropores, reducing the available surface for adsorption (Abd et al., 2021). Therefore, lower moisture levels free up adsorption sites, increasing adsorption efficiency. A significant reduction in volatile matter, from 75.2 % to 6.6 %, indicates the removal of organic compounds during activation, leaving a carbon-rich structure. As a consequence, a substantial increase in fixed carbon confirms successful carbonization and activation, critical for high surface area and adsorption performance (Mai et al., 2021). The ultimate result also confirms the enrichment of carbon during activation. B2 has slightly higher fixed carbon content than B1. Meanwhile, the slight increase in ash content reflects the concentration of inorganic residues after organic matter decomposition. The ash content of bamboo-derived biochar has significantly lower ash

content compared to coal-based carbons, and is within the lower range for wood-based activated carbons, making it competitive in terms of purity (Acevedo et al., 2020; Hong et al., 2021). It can be interpreted that there is not much difference between B1 and B2 in the aforementioned characteristics, despite being activated in two different environments. The difference is only reflected in the bulk density. The bulk density nearly doubles in activated carbon, which suggests a more compact and porous structure post-activation. This change is advantageous for applications requiring high adsorption capacity per unit volume (Acevedo et al., 2020). The lower bulk density of B2 indicates that the activation process in steam creates more porous structures within the material. This is consistent with findings from previous studies, where activation in CO₂ was shown to produce fewer mesoporous structures compared to activation in H₂O, due to the smaller molecular size and slower kinetics of CO₂ (Mai et al., 2021). Bamboo-derived biochar have a higher bulk density than most wood-based activated carbons (typically in the range of 250–500 kg/m³) (Čespiva et al., 2023). Minimal nitrogen content remains unchanged, suggesting nitrogen-containing groups are not significantly affected. Also, the potential of NO_x emissions during activation process is negligible (Khuong et al., 2022).

SEM techniques were used to closely observe the morphology of the bamboo-derived biochars. It is worth noting that SEM images only provide a relative representation of the char structure, as the images can vary depending on different zoom levels and angles. Fig. 2 represents two different angles and magnifications for the two types of bamboo-derived biochar after activation. At lower magnifications, the morphology of bamboo-derived biochar activated with steam shows greater porosity compared to bamboo-derived biochar activated with CO₂, which is consistent with the bulk density result. Additionally, the surface of the steam-activated charcoal appears relatively smoother, indicating the greater abrasive effect of steam. At higher magnifications, when zooming in on the surface of the two biochars, it is evident that the bamboo-derived biochar activated with steam exhibits more pronounced surface destruction, which contributes to the difference in porosity between the two bamboo biochars.

3.2. Adsorption by concentration

The adsorption capacity and efficiency of bamboo-derived biochar (B1 and B2) were evaluated at OTC concentrations ranging from 50 to 400 ppm (Fig. 3). Results indicated that at lower concentrations, the removal efficiency was higher due to an abundance of active adsorption sites relative to the available OTC molecules. However, at higher concentrations, the removal efficiency decreased as the adsorption sites became saturated. Notably, B2 demonstrated a slightly higher adsorption efficiency than B1 which is attributed to its optimized porosity and active surface area for OTC interaction.

3.3. Adsorption isotherm

The Langmuir, Freundlich, Temkin, and Redlich–Peterson adsorption models were applied to evaluate the adsorption process of oxytetracycline (OTC, initial concentration in the range of 50 – 400 ppm) on two types of bamboo biochar (CO₂-activated biochar and H₂O-activated biochar) (Fig. 4, Table 2). The analysis results indicate that each model exhibits varying degrees of fit, reflecting the distinct characteristics and adsorption mechanisms of each type of biochar.

The Freundlich isotherm, which accounts for heterogeneous surface adsorption, also provided strong fits for the data. B2 demonstrated superior performance with a Freundlich constant (K_f) of 8.876 L/mg, nearly double that of B1 (4.07 L/mg). The Freundlich exponent (n_f) values were 4.045 for B2 and 3.65 for B1, indicating that B2 offered a more homogeneous distribution of adsorption sites. Additionally, the higher correlation coefficient ($R^2 = 0.98$) for B2 compared to B1 ($R^2 = 0.93$) underscores its better suitability for Freundlich adsorption. These

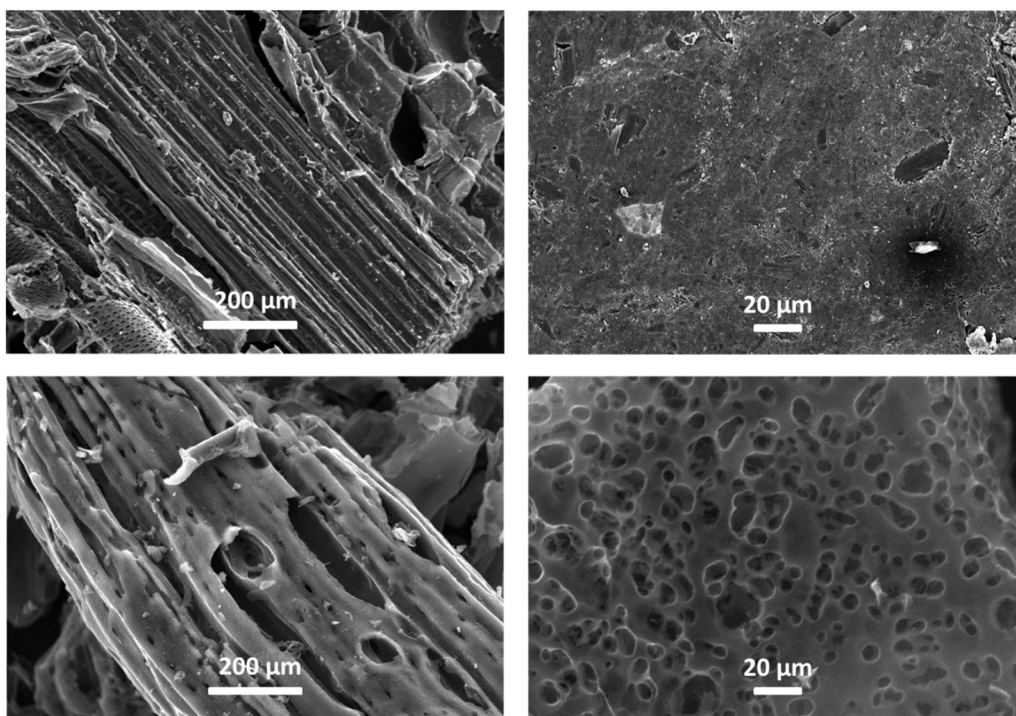


Fig. 2. SEM images of B1 (higher images) and B2 (lower images).

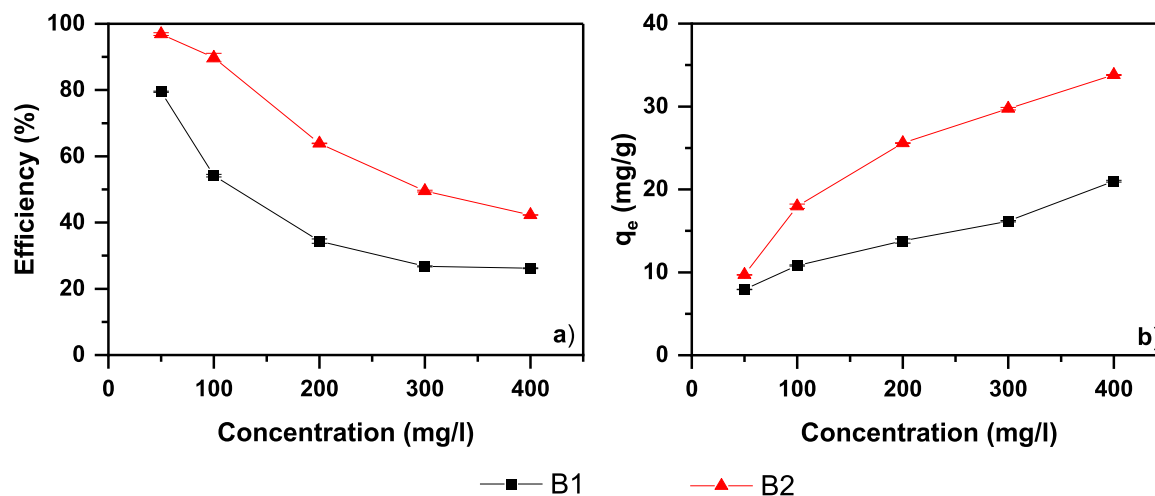


Fig. 3. a) Adsorption efficiency and b) Adsorption capacity of biochar.

results suggest that B2 provides more efficient and uniform adsorption behavior than B1.

The Langmuir isotherm assumes monolayer adsorption on a homogeneous surface. Experimental results indicate that H₂O-activated biochar exhibited a significantly higher maximum adsorption capacity ($q_{\max}$) of 34.31 mg/g compared to 21.51 mg/g for CO₂-activated biochar. Additionally, the Langmuir constant (K_L) for H₂O-derived biochar (0.076 L/mg) was greater than that for CO₂-activated biochar (0.024 L/mg), reflecting stronger adsorption affinity for oxytetracycline (OTC). Both biochars showed favorable adsorption behavior, as evidenced by the R_L values ranging from 0.454 to 0.094 for B1 and 0.208–0.031 for B2, at initial OTC concentrations (C_0) of 50–400 ppm. These results highlight that the Langmuir isotherm adequately describes the adsorption on both materials, with a higher adsorption capacity for H₂O biochar.

The Temkin isotherm, which emphasizes the role of adsorbate-

adsorbent interactions, yielded better fits for B2 ($R^2 = 0.98$) than for B1 ($R^2 = 0.83$). The Temkin constant (K_T) for B2 (3.94 L/g) was significantly higher than that for B1 (0.749 L/g), indicating stronger interactions between OTC and the B2 surface. Furthermore, the higher B_T value (4.74 J/mol) for B2 compared to B1 (3.3895 J/mol) suggests higher average adsorption energy, further confirming the stronger affinity of B2 for OTC.

The Redlich–Peterson (R-P) isotherm combines the features of Langmuir and Freundlich models, making it suitable for describing adsorption on heterogeneous surfaces. The R-P model exhibited the best fit for both biochars, with correlation coefficients of $R^2 = 0.998$ for B2 and $R^2 = 0.992$ for B1. The β parameter, which indicates the extent of homogeneity, was comparable between the two biochars (0.753 for B2 and 0.735 for B1), reflecting mixed adsorption mechanisms. Despite the higher R-P constant ($K_{RP} = 0.242$ L/g) for B1 compared to B2 (0.113 L/g), the overall adsorption performance of B2 remained superior due to

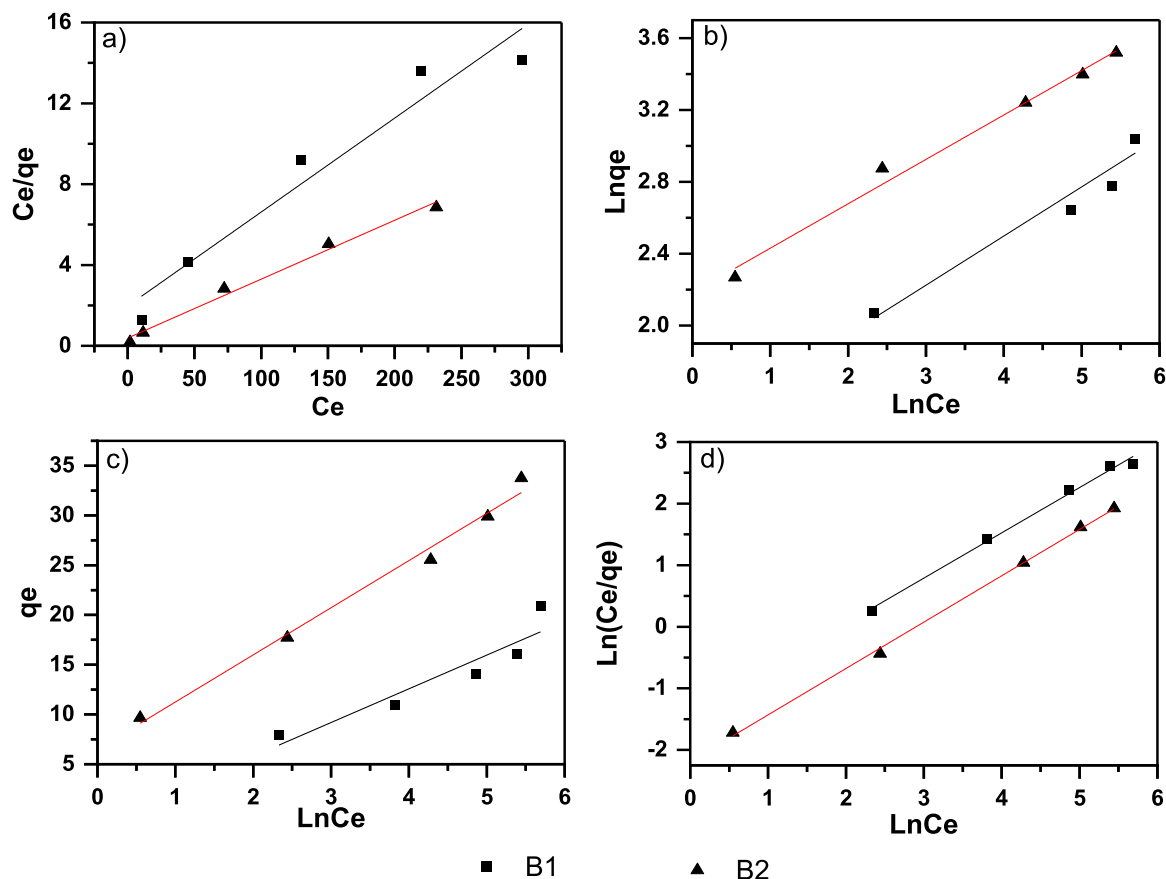


Fig. 4. a) Langmuir b) Freundlich c) Temkin và d) Redlich-Peterson of OTC adsorption on biochars.

its higher adsorption capacity. These findings suggest that while B2 is better for achieving maximum adsorption, B1 may excel under specific environmental conditions due to its balanced adsorption mechanisms.

The adsorption behavior of OTC on H_2O - and CO_2 -activated biochars was well-described by Langmuir, Freundlich, Temkin, and Redlich-Peterson isotherms. Among these, the R-P model provided the best fit, reflecting the heterogeneous nature of both materials. B2 demonstrated superior adsorption capacity and stronger adsorbate-adsorbent interactions across all models, making it a more efficient adsorbent for OTC removal. However, the flexibility of B1, as indicated by its ability to perform effectively under heterogeneous conditions described by the R-P model, highlights its potential for applications in complex environmental systems.

3.4. Adsorption over time - adsorption kinetics

The removal efficiency of oxytetracycline (OTC) by H_2O - and CO_2 -activated biochars demonstrated significant differences over time (Fig. 5). H_2O biochar achieved a rapid increase in removal efficiency, reaching 80 % within the first 30 minutes and stabilizing at 92 % after 120 minutes, with a maximum adsorption capacity (q_e) of 18.5 mg/g. In contrast, CO_2 biochar displayed a slower adsorption rate, achieving a maximum removal efficiency of 57 % at 60 minutes and stabilizing at 54 % after 120 minutes, with a maximum q_e of 11.3 mg/g. These results highlight the superior adsorption properties of H_2O biochar, attributed to its larger pore volume, higher surface area, and enhanced adsorbate-adsorbent interactions.

The adsorption kinetics of oxytetracycline (OTC) on CO_2 - and H_2O -activated biochars were analyzed using four common models: Pseudo-First-Order (PFO), Pseudo-Second-Order (PSO), Elovich, and Intra-Particle Diffusion (Fig. 6, Table 3). Each model provides unique

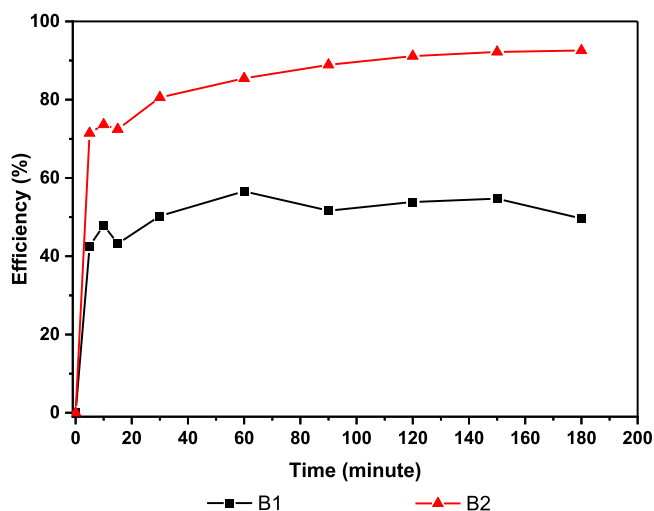


Fig. 5. OTC adsorption efficiency over time.

insights into the adsorption mechanism, and their applicability to this system was evaluated.

The PFO model, which assumes the adsorption rate is proportional to the difference between equilibrium adsorption capacity (q_e) and the adsorption capacity at time t , provided moderately good fits for both biochars. For B1, the calculated q_e was 10.29 mg/g, with an $R^2 = 0.947$ while for B2, q_e was 17.19 mg/g with $R^2 = 0.935$. However, the discrepancy between calculated q_e and experimental values (e.g. $q_e = 11.3$ mg/g for B1 and $q_e = 18.5$ mg/g for B2) indicates that PFO does not fully capture the adsorption behavior.

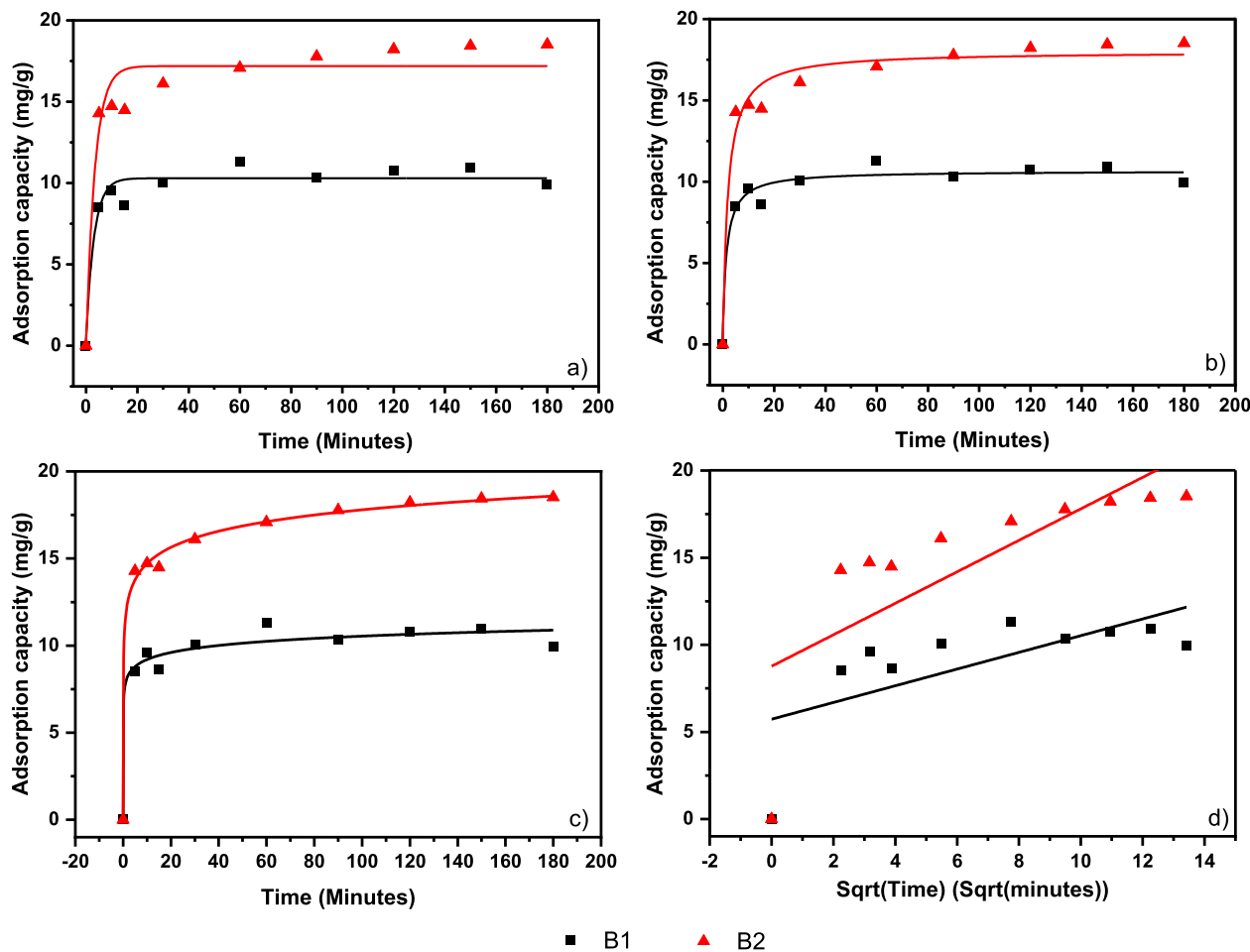


Fig. 6. a) Pseudo-first order kinetic, b) Pseudo-second order kinetic, c) Elovich model, d) Intra-particle diffusion kinetic model of biochars.

Table 3
Characteristics of raw bamboo and its activated carbon.

Characteristics		Raw bamboo	B1	B2
Moisture (wt%, ar)	M	9.5 ± 0.3	6.5 ± 0.2	6.3 ± 0.3
Bulk density (kg m ⁻³ , ar)	BD	260 ± 2	520 ± 25	470 ± 20
Proximate analysis (wt%, db)	V	75.2 ± 0.3	6.6 ± 0.2	6.1 ± 0.3
	A	1.9 ± 0.1	3.5 ± 0.1	3.7 ± 0.2
	FC	22.9 ± 0.4	89.9 ± 0.3	90.2 ± 0.5
Ultimate analysis (wt%, daf)	C	46.5 ± 0.2	86.5 ± 0.3	87.1 ± 0.3
	H	6.2 ± 0.2	5.7 ± 0.1	4.9 ± 0.1
	N	0.2 ± 0.1	0.2 ± 0.1	0.2 ± 0.1
	O	47.1 ± 0.5	7.6 ± 0.5	7.8 ± 0.5

ar: as received, db: dry basis, daf: dry-ash-free basis

The PSO model, which assumes chemisorption as the rate-limiting step, showed the best fit among all models, with $R^2 = 0.975$ for B2. The calculated q_e values, 10.67 mg/g for B1 and 18.00 mg/g for B2, were in excellent agreement with experimental results, confirming the chemisorption-dominated adsorption process. Furthermore, the adsorption rate constant (k_2) was higher for CO₂ biochar (0.064 g/mg.min) than for H₂O biochar (0.029 g/mg.min), reflecting differences in adsorption dynamics between the two materials.

The Elovich model often used for systems with heterogeneous surfaces, effectively captured the initial adsorption phase. The initial adsorption rates (α) and the desorption constant (β) differed significantly for B1 with higher values than B2, indicating a faster decline in adsorption rate over time for B1. Despite its utility in describing initial kinetics, the Elovich model had lower R² values (0.96 for B1 and 0.99

for B2), suggesting it was suitable for capturing the overall adsorption process.

The Intra-Particle Diffusion model was employed to evaluate the role of pore diffusion. The diffusion rate constant (k_p) was significantly higher for B2 (0.903 mg/g.min^{0.5}) compared to B1 (0.479 mg/g.min^{0.5}), reflecting faster intra-particle diffusion for B2. However, the high intercept (C) values for both biochars ($C=5.72$ mg/g for B1 and $C=8.77$ mg/g for B2) indicate that surface adsorption plays a dominant role, and intra-particle diffusion is not the sole rate-limiting step. The low R^2 values for this model (0.37 for B1 and 0.51 for B2) further confirm its limited applicability in describing the overall adsorption kinetics.

Overall, the PSO and Elovich models provided the best fit for the experimental data, highlighting chemisorption as the primary mechanism for OTC adsorption on the surface of both biochars. The superior performance of B2, with a higher q_e , stronger fits across all models, and faster diffusion rates, can be attributed to its larger surface area, better pore structure, and more effective functional groups. These results are consistent with prior studies, which also identified PSO as the best-fit model for biochar-based adsorption systems (Tran et al., 2024). Another study on bamboo sawdust-derived high surface area activated carbon demonstrated effective removal of paracetamol (PCT) from water (Wakejo et al., 2023). The maximum adsorption capacity predicted by the Langmuir model was 188.67 mg/g at an initial concentration of 120 mg/L. The adsorption followed both the Langmuir and Freundlich models, with the Redlich-Peterson model ($R^2 = 0.99$) providing the best fit, confirming monolayer adsorption on a heterogeneous surface. The adsorption process followed a pseudo-second-order kinetic model ($R^2 = 0.99$), indicating chemical adsorption as the

dominant mechanism. Moreover, this biochar after the optimized chemical modification using $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ and KOH , enhanced the surface area and functional properties of the biochar, the removal efficiency reached 94.07 % for acetaminophen (ACM) and 91.15 % for ciprofloxacin (CIP) under optimal conditions (Wakejo et al., 2024a). The adsorption process followed a pseudo-second-order kinetic model, suggesting chemisorption as the dominant mechanism. Tables 4 and 5

3.5. Effect of pH and pHpzc

The adsorption efficiency of oxytetracycline (OTC) on CO_2 - and H_2O -activated biochars was evaluated under varying pH conditions, ranging from pH 4 to pH 10. The point of zero charge (pHpzc) of B1 was determined to be 8.3, while that of B2 was 8.2. The pH of the solution plays a crucial role in influencing the surface charge of biochars and the speciation of OTC, both of which affect adsorption efficiency.

For B1, the adsorption efficiency exhibited a steep decline with increasing pH. At pH 4, the removal efficiency was approximately 50 %, which decreased significantly to less than 10 % at pH 10. This trend is closely linked to the surface charge of CO_2 biochar, which becomes negatively charged above its pHpzc. At pH values below 8.3, the biochar surface is positively charged, facilitating the adsorption of negatively charged OTC molecules (OTC^-). However, as the pH increases above the pHpzc, electrostatic repulsion occurs between the negatively charged biochar surface and OTC^- ions, leading to a marked decrease in adsorption efficiency. This strong dependence on pH highlights the limitations of B1 in alkaline environments.

In contrast, B2 demonstrated a relatively stable adsorption efficiency across the entire pH range, with values ranging from approximately 72 % at pH 4–78 % at pH 10. This stability indicates that the adsorption mechanism of B2 is not solely dependent on electrostatic interactions. The presence of abundant functional groups, such as hydroxyl and carboxyl groups, on the surface of B2 likely enables non-electrostatic interactions, such as hydrogen bonding and π - π interactions, which remain effective even in alkaline conditions. These interactions allow B2 to maintain high adsorption efficiency regardless of pH variations.

The observed trends in adsorption efficiency are consistent with the speciation of OTC in aqueous solutions. OTC exists in different ionic forms depending on pH: cationic (OTC^+) at $\text{pH} < 3.3$, zwitterionic ($\text{OTC}^\pm$) at $\text{pH} 3.3\text{--}7.5$, and anionic (OTC^-) at $\text{pH} > 7.5$. For B1, the repulsion between the anionic OTC^- species and the negatively charged biochar surface at $\text{pH} > 8.3$ reduces adsorption. In contrast, B2's stable performance suggests that its adsorption is driven by mechanisms beyond electrostatics, such as strong chemical interactions and surface functionality.

These findings have significant implications for practical

Table 4
Isotherm parameters for OTC adsorption on biochars.

Model	Constants and determination coefficients	B1	B2
Freundlich	n_f	3.65	4.045
	K_f (L/mg)	4.07	8.876
	R^2	0.93	0.98
Temkin	B_T (J/mol)	3.3895	4.74
	K_T (L/g)	0.749	3.94
	R^2	0.83	0.98
Langmuir	q_{max} (mg/g)	21.51	34.31
	K_L (L/mg)	0.024	0.076
	R^2	0.92	0.99
	R_L C_0 50	0.454	0.208
	R_L C_0 100	0.294	0.116
	R_L C_0 200	0.172	0.061
	R_L C_0 300	0.122	0.042
	R_L C_0 400	0.094	0.031
	K_{RP} (L/g)	0.242	0.113
Redlich–Peterson	β	0.735	0.753
	R^2	0.99	0.998

Table 5
Parameters of kinetic models for the adsorption of biochars for OTC.

Model	Parameters	B1	B2
Pseudo-First-Order (PFO)	q_e (mg/g)	10.294	17.189
	K_1 (1/min)	0.319	0.29
	R^2	0.95	0.93
Pseudo-Second-Order (PSO)	q_e (mg/g)	10.66	18.002
	K_2 (g/mg.min)	0.064	0.029
	R^2	0.97	0.98
Elovich	α (mg/g.min)	394557.4	7797.4
	β (g/mg)	1.71	0.74
	R^2	0.96	0.99
Intra-particle diffusion	K_p (mg/g.min ^{0.5})	0.479	0.903
	C	5.72	8.77
	R^2	0.37	0.51

applications. In real-world wastewater treatment, the pH of effluents can vary widely. B2 emerges as a more versatile and effective adsorbent for OTC removal due to its consistent performance across a broad pH range. In contrast, B1 may require pH adjustment to maximize adsorption efficiency, particularly in alkaline environments. However, the minimal effect of pH in general on the adsorption efficiency of oxytetracycline (OTC) for steam activated biochar, coupled with its consistently higher performance compared to CO_2 -activated biochar, can be explained by differences in activation methods and underlying adsorption mechanisms. Steam activation (H_2O) typically enhances biochar properties by increasing microporosity, specific surface area, and oxygen-containing functional groups such as $-\text{COOH}$, $-\text{OH}$, and $-\text{C}=\text{O}$. These groups facilitate chemical adsorption processes, such as hydrogen bonding and π - π interactions, which remain effective across varying pH levels. Consequently, the adsorption efficiency of B2 remains stable. In contrast, the electrostatic interactions limit the applicability of B1 in environments with fluctuating or alkaline pH. Fig. 7

3.6. Continuous adsorption

Two fixed-bed column experiments were conducted to evaluate the adsorption performance of two biochars, B1 and B2, under continuous flow conditions. Each column was packed with 10 g of biochar, and a 20 mg/L OTC solution was pumped through the columns at a flow rate of 1 L/h using a peristaltic pump operating at 60 rpm. The experiments were conducted under natural pH without adjustment. After reaching saturation (>90 % breakthrough), the columns were rinsed with 5 liters of distilled water at a constant flow rate, and the biochar was reused under the same initial conditions. SEM images of both biochars (B1 and B2) after OTC adsorption were captured at various magnifications and

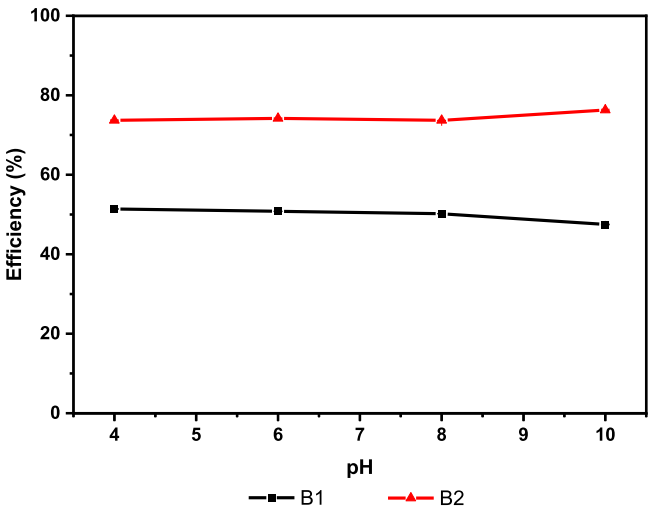


Fig. 7. Adsorption efficiency according to pH.

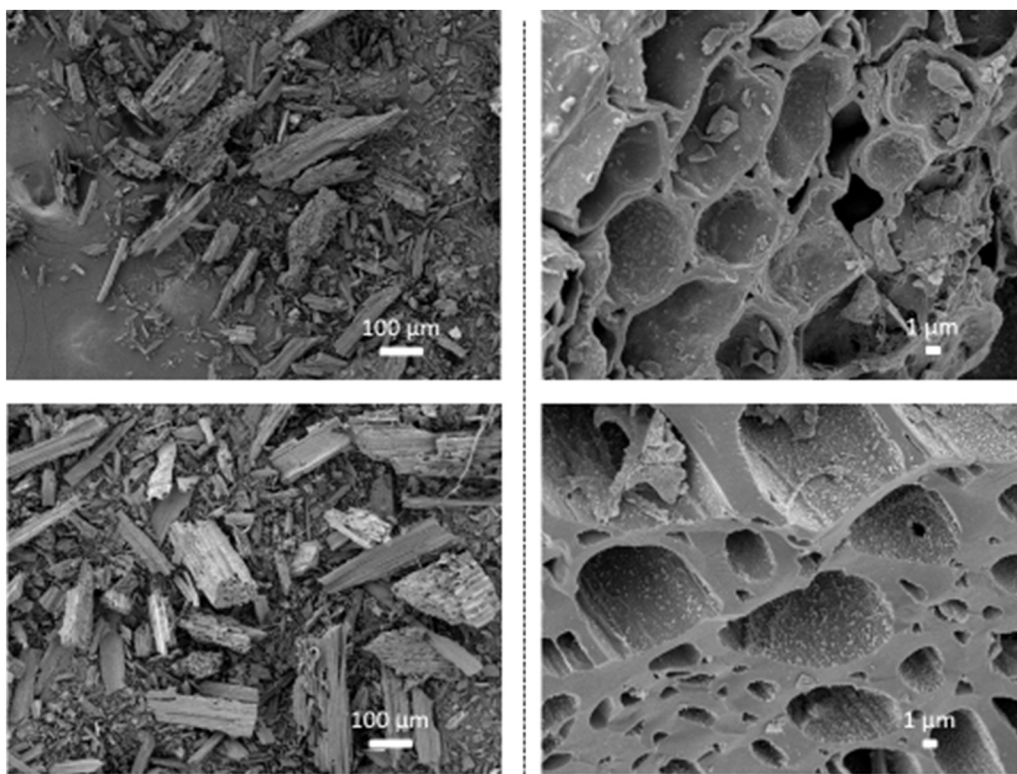


Fig. 8. SEM images of B1 (higher images) and B2 (lower images) after OTC adsorption.

locations (Fig. 8).

The morphology of the biochar after OTC adsorption in water shows no significant differences. However, at $3000\times$ magnification, numerous white spots are visible, indicating that OTC has strongly adhered to the surface and within the pores of the biochar. The adsorption results are summarized in Fig. 9.

The results indicate that in the fresh usage condition, sample B2 demonstrated superior adsorption performance compared to B1. Specifically, the C_e/C_0 value of B2 reached equilibrium at a lower level (~ 0.65) after 30 hours, whereas B1 stabilized at ~ 0.77 after 20 hours, indicating that B2 retained more OTC than B1. Under the recycle condition, B2 continued to outperform B1, with a stable C_e/C_0 value of 0.1, significantly lower than B1's 0.4. This result highlights the improved removal efficiency of OTC with increased contact time between the OTC molecules and the activated carbon. However, after regeneration, both samples exhibited a dramatic decline in performance, with C_e/C_0 values

increasing to ~ 0.8 – 0.9 , demonstrating that the regeneration method using distilled water was insufficient to restore the adsorption capacity of the materials. These findings confirm that B2 possesses a higher adsorption capacity than B1 under both fresh and recycled conditions, emphasizing the superior porous structure of B2's activated carbon. However, the current regeneration method is ineffective for both samples, resulting in low reusability after regeneration. Therefore, improvements in regeneration techniques or the development of novel adsorbent materials are necessary to enhance the efficiency of OTC removal from wastewater. The regeneration process using distilled water was insufficient to fully recover the adsorption capacity of both biochars. This reduced performance in reused biochars can be attributed to several factors, including the incomplete desorption of adsorbed OTC molecules, potential structural alterations or fouling of the biochar surfaces during the initial adsorption cycle may have further hindered the availability of active sites for subsequent adsorption.

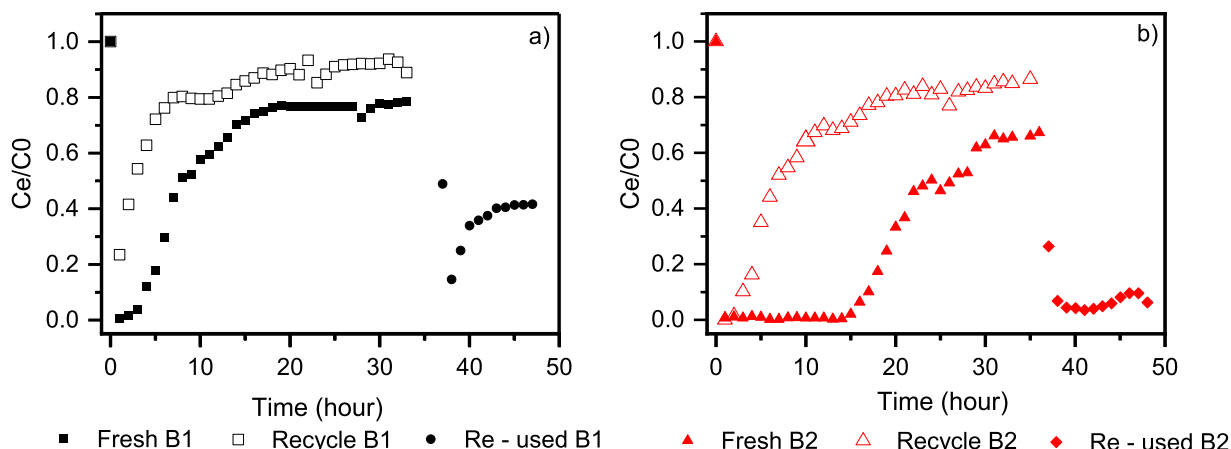


Fig. 9. The efficiency of OTC removal using a column adsorption model containing 10 g of biochar at a flow rate of 1 L/h: a) B1, b) B2.

The maximum adsorption capacities ($q_{\max}$) of biochars B1 and B2 for oxytetracycline (OTC) were evaluated in a continuous fixed-bed column system. Each column was packed with 10 g of biochar, and a 20 mg/L OTC solution was pumped at a flow rate of 1 L/h under natural pH conditions without adjustment. The breakthrough time (t_b) was determined as the time when the effluent concentration reached $C_e/C_0 = 0.8$, signifying column saturation. For fresh B1, the breakthrough occurred at 20 hours, yielding a maximum adsorption capacity of B1, $q_{\max} = 44$ mg/g. In contrast, fresh B2 exhibited a longer breakthrough time of 50 hours, resulting in a higher maximum adsorption capacity of B2, $q_{\max} = 100$ mg/g. The results showed that adsorption efficiency in column systems is superior to batch systems due to continuous flow, better mass transfer, and prolonged contact time. This result is consistent with several other studies. Wakejo et al. studied on tuneable functionalized biochar for pharmaceutical removal demonstrated high adsorption capacities for acetaminophen (ACM) and ciprofloxacin (CIP) (Wakejo et al., 2024b). In batch adsorption experiments, the maximum adsorption capacities predicted by the Langmuir model were 192.43 mg/g for ACM and 70.95 mg/g for CIP. In fixed-bed column adsorption, the biochar exhibited a maximum bed adsorption capacity of 172.48 mg/g for ACM and 147.67 mg/g for CIP, with higher removal efficiencies compared to batch studies.

These results demonstrate that B2 outperformed B1 in terms of adsorption capacity, likely due to superior structural properties, such as higher surface area, pore volume, or functional group density, which enhanced its affinity for OTC molecules. The findings underscore the critical role of biochar preparation and structural characteristics in optimizing adsorption performance in continuous flow systems. The evaluation of $q_{\max}$ provides a quantitative basis for comparing the adsorption efficiencies of B1 and B2, highlighting B2's potential for practical wastewater treatment applications. Further investigations into regeneration protocols and long-term stability are necessary to enhance the reusability and cost-effectiveness of these materials in real-world scenarios.

4. Conclusion

Among the two materials, B2 consistently exhibited superior adsorption performance, achieving higher adsorption capacities in both systems due to its enhanced porosity and functional group distribution. In batch experiments, B2 achieved a maximum adsorption capacity of 34.31 mg/g, compared to 21.51 mg/g for B1. In continuous column tests, B2 further demonstrated its efficiency with a breakthrough time of 50 hours and a maximum adsorption capacity of 100 mg/g, significantly surpassing B1's performance.

However, the study also highlights the limitations of the current regeneration process using distilled water, which was insufficient to restore the adsorption capacity of either material. After regeneration, both B1 and B2 exhibited significant performance declines, with C_e/C_0 values exceeding 0.8, indicating limited reusability. This finding underscores the need for optimizing regeneration techniques to enhance the practical viability of these adsorbents.

Overall, the results confirm the potential of bamboo biochars, particularly B2, as the effective and sustainable adsorbents for OTC removal. Future research should focus on developing more effective regeneration protocols and exploring modifications to improve the durability and reusability of bamboo biochars. These advancements will be critical for scaling up their application in wastewater treatment systems and addressing the growing challenge of antibiotic contamination in aquatic environments.

CRediT authorship contribution statement

Nguyen Hong Nam: Writing – review & editing, Writing – original draft, Visualization, Validation, Resources, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization. **Bui Thanh**

Phong: Software, Methodology, Investigation, Formal analysis. **Tran Thi Thu Huong:** Visualization, Software, Methodology, Investigation, Formal analysis. **Nguyen Thi Hong Hanh:** Methodology, Investigation, Formal analysis. **Le Phuong Thu:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work the authors used chatgpt.com in order to improve language and readability, with caution. After using this tool, the authors reviewed and edited the content as needed and took full responsibility for the content of the publication.

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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Data Availability

Data will be made available on request.

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