



Adsorption behavior of tetracycline and methylene blue onto microwave-assisted pine bark biochar

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ABSTRACT

The widespread occurrence of antibiotics and synthetic dyes in aquatic environments poses serious environmental challenges due to their persistence and potential ecological impacts. This study evaluated the adsorption performance of tetracycline (TC) and methylene blue (MB) onto pine bark-derived biochar prepared via microwave-assisted pyrolysis. Optimal adsorption was achieved at pH 7 for TC and MB, with equilibrium attained within 120 min. The adsorption kinetics of both pollutants were best described by the pseudo-second-order model, while the Langmuir isotherm provided the best fit to the equilibrium data, suggesting predominantly monolayer adsorption on homogeneous adsorption sites. The maximum adsorption capacities were 36.36 mg.g⁻¹ for TC and 163.93 mg.g⁻¹ for MB. These findings highlight the potential of microwave-assisted pine bark biochar as an efficient, sustainable, and cost-effective adsorbent for the removal of antibiotic and dye contaminants from aqueous systems.

Introduction

The increasing discharge of emerging contaminants into aquatic environments has become a major environmental concern worldwide. Among these contaminants, antibiotics and synthetic dyes are frequently detected in surface water, groundwater, and wastewater treatment plant effluents due to their extensive use in human activities and their incomplete removal by conventional treatment technologies. The persistence of these compounds in aquatic systems can lead to adverse ecological impacts, including toxicity to aquatic organisms, disruption of microbial communities, and deterioration of water quality [1].

Tetracycline (TC) is one of the most widely used broad-spectrum antibiotics in human medicine, veterinary practice, and aquaculture. Owing to its extensive consumption and relatively low metabolic degradation, a considerable fraction of administered tetracycline is excreted and subsequently released into the environment [2]. The continuous presence of tetracycline residues in water bodies has raised significant concerns regarding the development of antibiotic-resistant bacteria and antibiotic resistance genes [3,4]. Consequently, the effective removal of tetracycline from wastewater has attracted increasing research attention [5].

Methylene blue (MB) is a cationic dye extensively employed in textile, paper, pharmaceutical, and printing industries. Although methylene blue is often used as a model dye compound, its discharge into aquatic systems can reduce light penetration, hinder photosynthetic activity, and negatively affect aquatic ecosystems. Furthermore, prolonged exposure to methylene blue may pose health risks to humans and aquatic organisms [1]. Therefore, the development of efficient treatment technologies for the removal of both antibiotic and dye contaminants remains an important challenge in environmental remediation [6].

Among the available wastewater treatment technologies, adsorption has been recognized as one of the most effective and economically attractive approaches because of its operational simplicity, high removal efficiency, and applicability to a wide range of contaminants [7]. The performance of adsorption processes largely depends on the characteristics of the adsorbent material. In recent years, biochar derived from biomass residues has received considerable attention as a sustainable adsorbent due to its porous structure, abundant surface functional groups, low production cost, and environmental friendliness [8].

Pine bark is an abundant forestry by-product generated in large quantities worldwide. The conversion of pine bark into biochar not only provides a value-added application for biomass waste but also contributes to circular economy and sustainable resource management [9,10]. Moreover, microwave-assisted pyrolysis has emerged as an attractive alternative to conventional pyrolysis because it enables rapid volumetric heating, shorter processing times, reduced energy consumption, and enhanced development of porous structures. These advantages make microwave-assisted biochar a promising material for environmental applications, particularly in water treatment [11].

Although the physicochemical properties of pine bark-derived biochar have been reported previously, a comprehensive understanding of its adsorption behavior toward different classes of organic contaminants remains limited [12]. In particular, comparative investigations involving both antibiotic and dye pollutants are still scarce. Understanding the effects of operating parameters and adsorption mechanisms is essential for evaluating the practical applicability of biochar in wastewater treatment systems. Therefore, the present study aims to investigate the adsorption behavior of tetracycline and methylene blue onto microwave-assisted pine bark biochar. The effects of solution pH, contact time, and initial pollutant concentration were systematically evaluated. In

addition, adsorption kinetics and equilibrium isotherms were analyzed to elucidate the adsorption characteristics and possible adsorption mechanisms of the material toward these representative antibiotic and dye contaminants.

Experimental and research methods

Instruments and equipment:

UV-Vis spectrophotometer (Ultrospec 2000, UK); furnace (Nabertherm, Germany); analytical balance (Satorius, Germany); microwave oven (Toshiba, Japan); magnetic stirrer with heating function (IKA, Germany); pH meter (Inolab 730, Germany); Whatman filter paper (pore size 20–25 μm), stainless-steel sieves (125 and 212 μm mesh sizes), 10 mL polyethylene sample vials, and standard laboratory glassware.

Chemicals:

Tetracycline hydrochloride (TC; $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_8\cdot\text{HCl}$) supplied by Sigma-Aldrich, USA; methylene blue trihydrate (MB; $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}\cdot 3\text{H}_2\text{O}$; CAS No. 7220-79-3) supplied by China; sodium hydroxide (NaOH) and hydrochloric acid (HCl, 37%) supplied by Merck, Germany. All chemicals were of analytical grade and used without further purification.

Synthesis of Biochar from Pine Bark

The BCMAP biochar used in this study was prepared from pine bark via conventional pyrolysis followed by microwave-assisted activation, as described in our previous study [12]. Briefly, pretreated pine bark particles (125–212 μm) were pyrolyzed under oxygen-limited conditions at 600°C with a heating rate of 10°C min^{-1} and a residence time of 2h. After cooling to room temperature, the obtained biochar was dried at 80°C for 24 h and subsequently subjected to microwave-assisted activation operated at 800 W for 10 min. Detailed synthesis procedures and physicochemical characterization data are provided elsewhere [12]. The prepared material was used directly for the adsorption experiments described herein.

The main physicochemical characteristics of BCMAP relevant to its adsorption performance are summarized here. SEM analysis revealed a porous and heterogeneous carbon structure. The BET results showed a high specific surface area of 592.32 $\text{m}^2\cdot\text{g}^{-1}$ and a total pore volume of 0.197 $\text{cm}^3\cdot\text{g}^{-1}$, confirming the well-developed porosity of the material. The pH_{pzc} was 8.3, indicating that the surface is positively charged below this value and negatively charged above it. FTIR analysis confirmed the presence of key functional groups such as $-\text{OH}$, $\text{C}=\text{O}$, and $\text{C}-\text{O}$, which are responsible for interactions with organic molecules [12].

Batch Adsorption Experiments

The adsorption performance of BMAP toward tetracycline (TC) and methylene blue (MB) was evaluated through batch experiments. Briefly, 0.1000 g of BMAP was added to 50 mL of TC or MB solution and shaken at 150 rpm for a predetermined time. After adsorption, the suspensions were filtered, and the residual concentrations of TC and MB were determined using a UV–Vis spectrophotometer at 355 and 664 nm, respectively. All experiments were performed in duplicate, and the average values were reported.

The effects of pH (2–10), contact time (10–240 min), and initial adsorbate concentration (20–100 mg.L⁻¹ for TC and 100–500 mg.L⁻¹ for MB) were investigated using a one-factor-at-a-time approach while maintaining all other experimental conditions constant.

Adsorption efficiency H (%) and the adsorption capacity q_e (mg.g⁻¹) was calculated using Equation:

$$H = \frac{C_0 - C_e}{C_0} \times 100 \quad q_e = \frac{(C_0 - C_e) \times V}{m}$$

Where: C_0 , C_e are the initial concentration and the remaining concentration after adsorption in the solution (mg.L⁻¹), respectively; m is the mass of the material used (g); V is the volume of the solution (L).

Results and discussion

Investigation of factors affecting the adsorption of TC and MB onto BMAP

The solution pH significantly influences the adsorption capacity by altering both the surface charge of the biochar and the speciation of TC molecules [13]. As shown in the results, the removal efficiency of TC onto BMAP increased sharply from 26.5% at pH 2 to a peak of 92.3% at pH 7, before declining to 64.5% at pH 10. The low adsorption efficiency at acidic conditions (pH 2–4) is attributed to the strong electrostatic repulsion between the positively charged biochar surface (pH < pH_{pzc} = 8.3) and the cationic TC species (TC⁺). Furthermore, the abundance of H⁺ ions competes with TC for active sites, further hindering the process. As the pH approached the pH_{pzc}, the removal efficiency reached its peak at pH 7. In this near-neutral range, TC exists primarily in zwitterionic (TC⁰) and anionic (TC⁻) forms. The high removal rate suggests that non-electrostatic forces, particularly π - π electron donor-acceptor interactions promoted by the highly aromatic structure of the BMAP and hydrogen bonding are the dominant mechanisms [14]. However, at pH 10, the efficiency dropped to 68.5%. At this stage, the pH exceeds the pH_{pzc} (8.3), causing the biochar surface to become negatively charged. The resulting electrostatic repulsion between the anionic surface and the TC⁻ species leads to the observed decrease in adsorption performance [15]. In conclusion, the BMAP exhibits optimal performance in the pH range of 7.0, making it highly effective for treating TC-contaminated water in near-neutral environments [16].

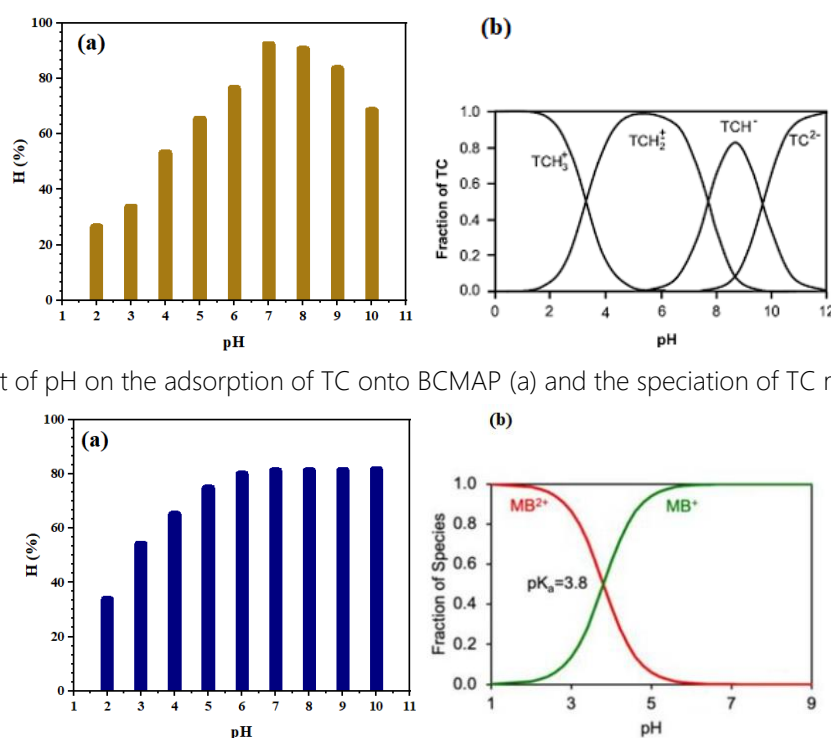


Fig 1: Effect of pH on the adsorption of TC onto BMAP (a) and the speciation of TC molecules (b)

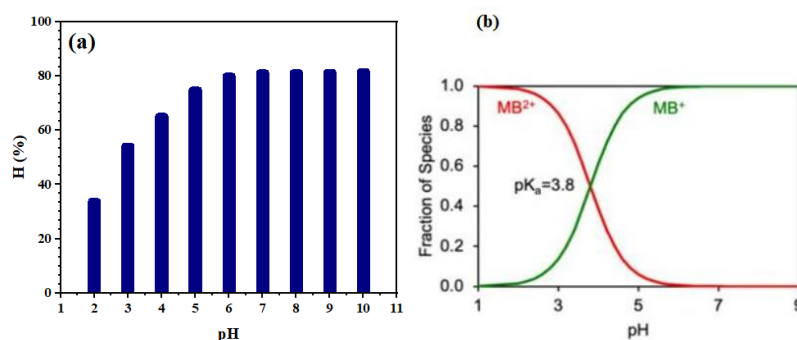


Fig 2: Effect of pH on the adsorption of MB onto BMAP (a) and the speciation of MB molecules (b)

The solution pH significantly influenced the adsorption of methylene blue (MB) onto BCMAP, as shown in Fig 2. The removal efficiency increased markedly from 33.8% at pH 2 to 81.2% at pH 7, after which only negligible changes were observed. The enhancement in adsorption performance with increasing pH can be attributed to changes in the surface charge of the biochar and the interactions between the adsorbent and MB molecules [17]. At acidic pH values, the BCMAP surface becomes highly protonated due to the abundance of H^+ ions in solution. Since methylene blue is a cationic dye, electrostatic repulsion between the positively charged adsorbent surface and MB molecules hinders adsorption. In addition, H^+ ions compete with MB molecules for available adsorption sites, resulting in lower removal efficiencies [18]. As the pH increased from 2 to 6, the concentration of H^+ ions decreased, reducing competition for active sites and facilitating the adsorption of MB. Simultaneously, the surface functional groups of BCMAP gradually deprotonated, generating negatively charged sites that enhanced electrostatic attraction toward the cationic dye molecules. Consequently, the adsorption efficiency increased substantially [19]. When the pH was further increased from 6 to 10, only a marginal improvement in removal efficiency was observed, with values remaining nearly constant at approximately 80–82%. This plateau

indicates that most accessible adsorption sites had already been occupied and that adsorption equilibrium was approached. The minimal increase in adsorption at higher pH values suggests that mechanisms other than electrostatic attraction, such as π - π interactions, pore filling, and hydrogen bonding, also contributed to the adsorption process [20]. FTIR analysis confirmed the presence of key functional groups such as $-OH$, $C=O$, and $C-O$, which can participate in hydrogen bonding and electrostatic interactions with organic molecules. In addition, the aromatic carbon framework of BCMAP provides abundant π -electron-rich domains, enabling strong π - π electron donor-acceptor interactions with the aromatic rings of organic molecules such as MB and TC. These π - π interactions, together with hydrogen bonding and electrostatic attraction, are expected to play a key role in the overall adsorption mechanism [12].

Although the highest removal efficiency (81.7%) was obtained at pH 10, the difference compared with pH 7 (81.2%) was negligible. Therefore, pH 7 was selected as the optimal operating pH for subsequent experiments because it provided high adsorption efficiency while avoiding the need for excessive pH adjustment. Moreover, neutral pH conditions are more environmentally relevant and economically favorable for practical wastewater treatment applications.

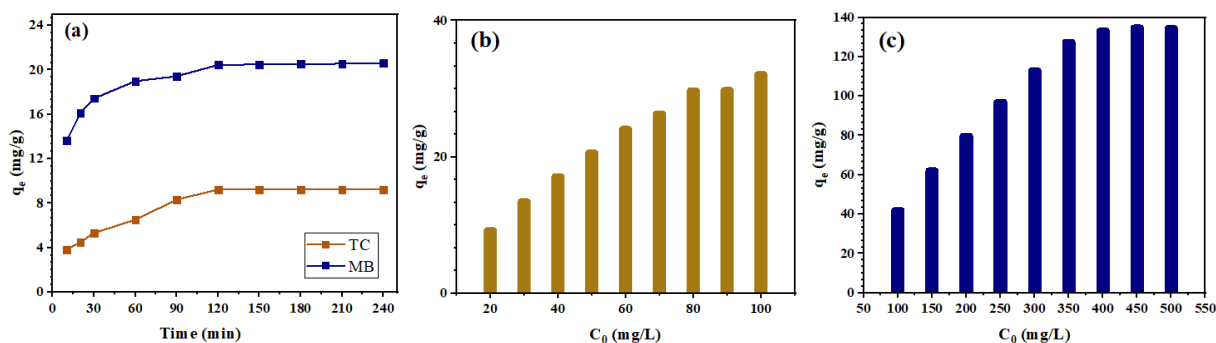


Fig 3: Effects of contact time on TC and MB adsorption (a) and initial concentration on TC (b) and MB (c) adsorption onto BCMAP.

As shown in Fig. 3a, the adsorption capacities of BCMAP toward TC and MB increased rapidly during the initial stage and gradually approached equilibrium. For TC, equilibrium was reached after approximately 120 min with an adsorption capacity of 9.23 mg.g^{-1} , whereas MB exhibited a higher adsorption capacity of 20.64 mg.g^{-1} under the same conditions. The rapid initial adsorption was attributed to the abundance of available active sites and the strong concentration gradient between the solution and adsorbent surface [21]. As adsorption progressed, the number of vacant sites decreased, resulting in a slower adsorption rate until equilibrium was established [20,22]. The higher adsorption affinity of

BCMAP for MB than for TC may be attributed to MB's cationic nature and smaller molecular size, which facilitate stronger electrostatic and π - π interactions with the biochar surface. In contrast, the more complex molecular structure of TC may experience greater steric hindrance during adsorption [23,24]. Therefore, 120 min was selected as the optimum contact time for subsequent experiments.

The effect of the initial concentration on the adsorption capacity of pine bark biochar toward TC and MB is presented in Fig 3b,c. As the initial concentration increased, the equilibrium adsorption capacity (q_e) increased markedly for both contaminants. For TC, q_e

increased from 9.23 to 32.11 mg.g⁻¹ when the initial concentration increased from 20 to 100 mg.L⁻¹. Similarly, the adsorption capacity for MB increased from 41.98 to 134.56 mg.g⁻¹ over the concentration range of 100–500 mg.L⁻¹. The increase in adsorption capacity can be attributed to the enhanced concentration gradient between the aqueous phase and the adsorbent surface, which provides a stronger driving force for mass transfer and increases the probability of interactions between pollutant molecules and available adsorption sites [25]. At lower concentrations, a large proportion of adsorption sites remained unoccupied, whereas increasing pollutant concentration promoted greater utilization of the porous structure and surface functional groups of the biochar [24]. Notably, the adsorption capacity of MB approached a plateau at concentrations above 400 mg.L⁻¹, suggesting progressive saturation of the available adsorption sites. A similar trend, although less pronounced, was observed for TC at higher concentrations. These results indicate that the adsorption process is concentration dependent and that the biochar exhibits a higher adsorption affinity and capacity toward MB than TC under the investigated conditions [24,26].

Adsorption Kinetics Study

Adsorption kinetics were investigated to elucidate the adsorption rate and mechanism of TC and MB onto BCBMAP. The experimental data were analyzed using the pseudo-first-order and pseudo-second-order kinetic models. The kinetic parameters were obtained from the corresponding linearized plots and are summarized in Table 1.

Table 1: Adsorption kinetic parameters for the TC and MB adsorption onto BCBMAP

Kinetic models	Kinetics model parameters		
		TC	MB
Pseudo-first-order	k_1	0.0235	0.0131
$\lg(q_e - q_t) = \lg q_e - \frac{k_1 \times t}{2.303}$	$q_{e, \text{cal}}$	6.05	21.23
	R^2	0.8509	0.8952
	$q_{e, \text{exp}}$	9.23	20.64
Pseudo-second-order	k_2	0.0004	0.075
$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \left(\frac{1}{q_e}\right) \times t$	$q_{e, \text{cal}}$	10.36	21.23
	R^2	0.9941	0.9998

q_e/q_t (mg.g⁻¹): adsorption capacities at equilibrium/at time t , respectively;

k_1 (min⁻¹)/ k_2 (g.mg⁻¹.min⁻¹): respective rate constants of studied kinetics models.

For both adsorbates, the pseudo-second-order model provided a significantly better fit to the experimental data than the pseudo-first-order model, as evidenced by the higher correlation coefficients ($R^2 = 0.9941$ for TC and 0.9998 for MB). For TC adsorption, the calculated

equilibrium adsorption capacity obtained from the pseudo-second-order model ($q_{e, \text{cal}} = 10.36$ mg.g⁻¹) was closer to the experimental value ($q_{e, \text{exp}} = 9.23$ mg.g⁻¹) than that predicted by the pseudo-first-order model ($q_{e, \text{cal}} = 6.05$ mg.g⁻¹). Similarly, for MB adsorption, the pseudo-second-order model yielded a calculated adsorption capacity ($q_{e, \text{cal}} = 21.23$ mg.g⁻¹) in good agreement with the experimental value ($q_{e, \text{exp}} = 20.64$ mg.g⁻¹). In contrast, the pseudo-first-order model exhibited lower correlation coefficients ($R^2 = 0.8509$ for TC and 0.8952 for MB), indicating a poorer description of the adsorption process.

The excellent fit of the pseudo-second-order model suggests that the adsorption rate was primarily governed by the availability of active adsorption sites and the interactions between pollutant molecules and functional groups on the BCBMAP surface [27]. These findings indicate that BCBMAP possesses strong adsorption affinity toward both TC and MB and that the adsorption process is predominantly controlled by surface-related interactions rather than simple diffusion in the aqueous phase [26].

Adsorption Isotherm Study

Adsorption isotherms were investigated to evaluate the equilibrium behavior of TC and MB on BCBMAP. The experimental data were analyzed using the Langmuir and Freundlich isotherm models. Isotherm experiments were conducted by varying the initial adsorbate concentration (20–100 mg.L⁻¹ for TC and 100–500 mg.L⁻¹ for MB), whereas the adsorbent dosage, pH, and contact time were maintained at the optimized conditions determined previously. The regression equations and corresponding parameters of the Langmuir and Freundlich isotherm models are presented in Table 2.

Table 2: Adsorption isotherm parameters for the TC and MB adsorption onto BCBMAP

Isotherm models	Isotherms model parameters		
		TC	MB
Langmuir $\frac{C_e}{q_e} = \frac{C_e}{q_{\text{max}}} + \frac{1}{q_{\text{max}} \times K_L}$	q_m	36.36	163.93
	K_L	0.1747	0.0261
	R^2	0.9937	0.9898
	K_F	8.49	19.96
Freundlich $\lg q_e = \lg K_F + \frac{1}{n} \times \lg C_e$	$1/n$	0.3916	0.4389
	R^2	0.9770	0.9031

q_e/q_{max} (mg.g⁻¹): equilibrium/maximum adsorption capacity of adsorbent; K_L (L.mg⁻¹): Langmuir constant; K_F (mg.g⁻¹): Freundlich constant; C_e (mg.L⁻¹): equilibrium concentration of adsorbate.

The Langmuir model exhibited superior fitting performance for both adsorbates, with correlation coefficients ($R^2 = 0.9937$ for TC and 0.9898 for MB)

markedly higher than those obtained from the Freundlich model. This result suggests that the adsorption process was dominated by adsorption onto a finite number of energetically comparable sites and that the adsorbate–surface interactions were adequately described by the Langmuir assumption [12]. The maximum adsorption capacities predicted by the Langmuir model reached 36.36 mg.g⁻¹ for TC and 163.93 mg.g⁻¹ for MB, highlighting the substantial adsorption potential of BCMAP toward both contaminants. The markedly higher capacity observed for MB indicates a more efficient occupation of available adsorption sites under the investigated conditions [24]. Furthermore, the Freundlich constants ($1/n < 1$) indicate favorable adsorption behavior and suggest the presence of surface heterogeneity. Nevertheless, the lower fitting accuracy of the Freundlich model implies that surface heterogeneity played a secondary role compared with the adsorption equilibrium represented by the Langmuir model [23]. Overall, the isotherm analysis demonstrates that the adsorption equilibrium of both TC and MB on BCMAP was governed primarily by site-limited adsorption, with the Langmuir model providing the most appropriate representation of the experimental data [6].

Conclusion

This study demonstrated the potential of microwave-assisted pine bark biochar as an effective adsorbent for the removal of TC and MB from aqueous solutions. Adsorption performance was strongly influenced by solution pH, contact time, and initial pollutant concentration, with optimal adsorption observed at pH 7 for TC and MB. Equilibrium was achieved within 120 min, indicating a relatively rapid adsorption process. Kinetic analyses revealed that the pseudo-second-order model provided the best description of the adsorption behavior, whereas the Langmuir isotherm showed the highest fitting accuracy, suggesting predominantly monolayer adsorption on a finite number of adsorption sites. The maximum adsorption capacities were 36.36 mg.g⁻¹ for TC and 163.93 mg.g⁻¹ for MB, demonstrating a notably higher affinity of BCMAP toward MB. Overall, the results highlight the feasibility of converting pine bark waste into a value-added biochar adsorbent and support its potential application in sustainable wastewater treatment systems for the removal of pharmaceutical and dye contaminants.

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