Applied Environmental Research
- RESEARCH FOR SUSTAINABLE PLANET -

Research Article

Ball-Milled Biochar from Waste Bamboo Chopsticks: A Potential Adsorbent for Methylene Blue Removal

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Abstract

Waste bamboo chopsticks encounter limited recycling. Several treatment procedures necessitate sophisticated technologies and intricate processes that need more practicality for local-scale production, leading to substantial challenges in effectively repurposing waste from disposable bamboo chopsticks. In this research, waste bamboo chopsticks were used as raw materials for the production of ball-milled biochar (BM-WBCB). FTIR, SEM, and BET were among the numerous analytical methods used to describe the BM-WBCB. The outcomes showed that the surface was made up of hollow, porous structures. In addition, the pHpzc value of the biochar was 8.7, and it had a BET-specific surface area (273.11 m^2 g^{-1}). The study also investigated ball-milled biochar's adsorption capacity and efficiency in removing MB from aqueous solutions. Batch adsorption experiments were conducted under various experimental conditions, such as initial dye concentration, contact time, pH, and adsorbent dose, to evaluate the adsorption kinetics, equilibrium, and thermodynamics of the adsorption process. The results showed the optimal adsorption conditions, such as pH solution, MB concentration, dose of BM-WBCB, and contact time at 10, 30 mg L^{-1}, 0.3 mg, and 60 min, respectively. With a maximal adsorption capacity of 4.2 mg g^{-1}, monolayer adsorption was shown by the fit of the adsorption isotherm data to the Langmuir isotherm model. It was demonstrated that the kinetics of biochar adsorption may be accurately modeled using the linear pseudo-second-order kinetic model.

ARTICLE HISTORY

Received: 13 Jul. 2023
Accepted: 8 Jan. 2024
Published: 23 Feb. 2024

KEYWORDS

Adsorption;
ball-milled Biochar;
Methylene blue removal;
Potential adsorbent;
Waste bamboo chopstick

Introduction

Water pollution is a pressing global issue that has gained increasing attention due to its detrimental impact on ecosystems and humans [1]. The contamination of water bodies by various pollutants, including industrial waste, agricultural runoff, and domestic effluents, has resulted in severe environmental consequences [2]. Among these pollutants, synthetic dyes have emerged as a significant concern due to their wide use in industries such as textiles, paper, and pharmaceuticals [3]. These dyes not only affect water aesthetics but can also be toxic to aquatic life, potentially disrupting the balance of ecosystems [4].

Methylene blue (MB), a cationic dye belonging to the thiazine class, is commonly used in numerous industries for coloring [5]. Its release into water bodies through effluents has raised concerns due to its persistence, potential toxicity, and carcinogenic properties. Therefore, developing efficient and eco-friendly methods for removing MB from contaminated water sources is paramount. Various decolorization treatment methods have been developed over the past decades, including physical, chemical, and biological techniques [6–7]. Adsorption is the preferred method, producing the best results among various color removal processes [8]. It is

https://doi.org/10.35762/AER.2024004

a well-known equilibrium separation technique that effectively decontaminates water.

Many approaches have been recently studied to develop cheaper and more effective adsorbents. Several researchers have proposed many low-cost adsorbents, including natural materials, biosorbents, and waste materials from agriculture and industry, for use as adsorbents to remove dyes from solutions [6, 9–10]. The utilization of low-cost and sustainable adsorbents holds significant potential advantages. They present an economically viable approach to water treatment in developing nations and other resource-constrained environments, all while upholding environmental sustainability.

Biochar, a carbonaceous material produced through biomass pyrolysis, has shown remarkable potential as an adsorbent for wastewater treatment due to its unique physicochemical properties [11–12]. Furthermore, biochar modification through mechanical treatment, such as ball milling, has shown potential in enhancing its adsorption capacity by increasing surface area and creating more accessible active sites [13–14]. Additionally, producing biochar from waste biomass presents a valuable opportunity to address waste management and environmental concerns [15]. Waste bamboo chopsticks, a prevalent commodity resulting in substantial waste generation, can serve as an ideal precursor for biochar synthesis.

The study will investigate the adsorption performance of ball-milled biochar for the removal of MB from contaminated water sources. The main objectives of the research are to evaluate the influence of ball milling on the physicochemical properties of biochar, determine the optimal adsorption conditions for MB removal, and explore the adsorption kinetics and isotherms governing the process. The findings of this study will contribute to the development of efficient and sustainable methods for water purification, with potential implications for addressing the challenges posed by water pollution.

Materials and methods

1) Materials procurement and collection

This investigation used disposable bamboo chopsticks obtained from Vietnamese restaurants and street foods. Methylene Blue ($C_{16}H_{18}ClN_3S$) was purchased from Xilong Chemical Co., Ltd., China. The reagent solutions were made with deionized (DI) water, and all of the compounds were of the analytical grade.

2) Preparation of adsorbent

After being collected, the waste bamboo chopsticks were cleaned many times to eliminate dirt before being slowly pyrolyzed for 120 min at 500°C in a specialized oven to produce biochar. Then, the biochar was ground to 0.5 to 1 mm particles size. To prepare ball-milled biochar, 10 g of the pristine biochar was mechanically activated in a planetary ball mill (RETSCH, PM 100 CM, USA) using a 120 mL capacity stainless steel grinding jar and 25 stainless steel grinding balls with 10 mm diameter. The ball milling was carried out for 10 min at 500 rpm. After milling, ball-milled biochar sample was labeled as BM-WBCB and used in adsorption experiments.

3) Analysis of adsorbent characterization

Several methods were employed to analyze the physicochemical properties of the BM-WBCB. The internal geometrical structure and surface morphology were examined using a Scanning Electron Microscope (SEM, Thermo Fisher Scientific, USA). The Fourier Transform Infra-Red (FTIR, Thermo Fisher Scientific, Nicolet iS5, USA) spectrometer was utilized to investigate the surface chemistry of adsorbent. Additionally, the BET surface area was determined by employing a Surfer gas adsorption porosimeter (Thermo Fisher Scientific, Surfer, USA), which involved nitrogen gas adsorption-desorption at a temperature of -196°C.

The pH drift method was used to estimate the BM-WBCB's pH at the point of zero charge (pHpzc) [16]. Initially, the pH values (pHi) of a 0.1 M NaCl solution were adjusted within the range of 2 to 12 by adding either 0.1 M HCl or 0.1 M NaOH. Subsequently, 1 g of BM-WBCB and 50 mL of the 0.1 M NaCl solution were combined in an Erlenmeyer flask. The mixture was then agitated using a rotary shaker at room temperature with a shaking frequency of 120 rpm for 24 hours. Afterward, the mixture was filtered through filter paper, and the resulting solution's final pH (pHf) was measured using a pH meter. Finally, the pHpzc was determined by plotting the difference in pH ($\triangle$pH = pHf - pHi) against the pHi. The pHpzc was identified as the intersection of the X-axis and the plotted curve.

4) Adsorption experiments

The efficiency of removing MB from an aqueous solution was investigated through batch experiments. Various reaction conditions were explored to determine their influence on MB removal efficiency. These conditions included adjusting the pH of the solution (ranging from 4 to 12), the contact time (ranging from 0 to 90 min), the dosage of adsorbent (ranging from 0.1 to 0.5 g), and the initial concentration of the MB solution (ranging from 10 to 50 mg L^{-1}). The adsorption studies were conducted using 50 mL of MB solution in Erlenmeyer flasks placed on a shaker at 120

rpm and a temperature of 30±2°C. Following the adsorption process, the adsorbent was separated from the suspension using filter paper. The filtrate concentration was then analyzed using a UV-Vis spectrophotometer. The instrument, a Thermo Fisher Scientific Evolution 350 (USA), was set to the wavelength of maximum absorbance for MB (665 nm). All experiments were performed three times.

The equilibrium amount of adsorbate, the adsorbate amount at a specific time, and the efficiency of MB removal were all determined using mathematical equations. The equations are Eq. 1–3.

The adsorption capacity at equilibrium:

$$q_e = \frac{C_0 - C_e}{m} \times V \quad \text{(Eq. 1)}$$

The adsorption capacity at a particular time:

$$q_t = \frac{C_0 - C_t}{m} \times V \quad \text{(Eq. 2)}$$

Removal efficiency:

$$H\% = \frac{C_0 - C_e}{C_0} \times 100 \quad \text{(Eq. 3)}$$

The following are the symbols used in the Eq. 1–3:

- q_e: adsorption capacity at equilibrium (mg g^{-1})
- q_t: adsorption capacity at a particular time (mg g^{-1})
- C_0: initial MB solution concentration (mg L^{-1})
- C_e: equilibrium MB solution concentration (mg L^{-1})
- C_t: MB solution concentration (mg L^{-1}) at time t
- V: volume of solution (L)
- m: mass of the adsorbent (g)

5) Adsorption isotherms

The Langmuir and Freundlich adsorption isotherms were used to investigate the equilibrium adsorption performance of BM-WBCB. The linear forms of the isotherm models' regression equations are given in Eq. 4–5.

Langmuir adsorption isotherm model:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}} \quad \text{(Eq. 4)}$$

Freundlich adsorption isotherm model:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad \text{(Eq. 5)}$$

The following are the symbols used in the Eq. 4–5:

- Ce (mg L^{-1}): equilibrium concentration of the MB
- qe (mg g^{-1}): adsorption capacity at equilibrium
- qmax (mg g^{-1}): maximum adsorption capacity
- KL (L mg^{-1}): Langmuir constant
- KF [(mg g^{-1})/(mg L^{-1})1/n]: Freundlich constant
- n (dimensionless): Freundlich intensity parameter.

6) Adsorption kinetics

Regarding adsorption kinetics, the pseudo-first-order (PFO) and pseudo-second-order (PSO) models were used to study the kinetics of MB adsorption onto the BM-WBCB. Eq. 6-7 present PFO and PSO as follows:

PFO kinetic model:

$$q_t = q_e\left(1-\exp^{-k_1 t}\right) \quad \text{(Eq. 6)}$$

PSO kinetic model:

$$q_t = \frac{q_e^2 k_2 t}{1+q_e k_2 t} \quad \text{(Eq. 7)}$$

The following are the symbols used in the Eq. 6-7:

- q_e (mg g^{-1}): adsorption capacity at equilibrium
- q_t (mg g^{-1}): adsorption capacity at a specific time t (min).
- k_1 (min^{-1}): PFO rate constant
- k_2 (g mg^{-1} min^{-1}): PSO rate constant

Results and discussion

1) Characterization of BM-WBCB

1.1) FTIR spectra of adsorbent

Figure 1 shows the FTIR spectra of the BM-WBCB. The absorption spectrum in the range of 3,500–3,300 cm^{-1} exhibited a dominant peak at approximately 3,428 cm^{-1}, attributed to the vibrational motion of O-H groups present in alcohols, phenols, and carboxylic acids, as observed in cellulose and lignin. Consequently, the adsorption of MB dye onto BM-WBCB was primarily facilitated by phenolic and acidic functional groups. Additionally, the absorption band at 1,585 cm^{-1} is characteristic of the stretching vibration of C-C bonds in aromatic rings. The peak at 1,118 cm^{-1} was attributed to the stretching vibration of C-O bonds from the secondary alcohol group. Another absorption spectrum was detected in the 877–752 cm^{-1} range, indicating C-H bending vibrations.

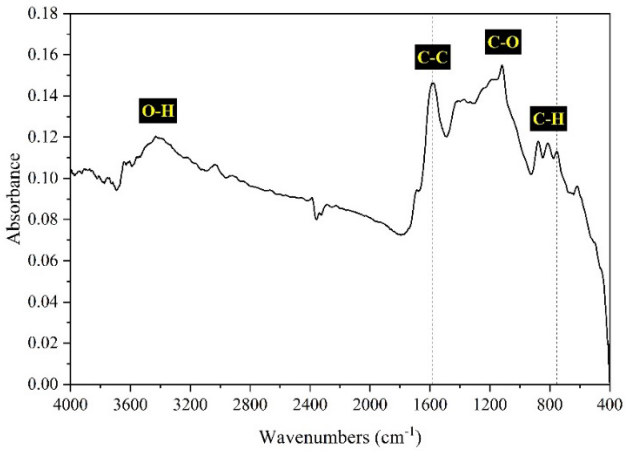

Figure 1 FTIR spectra of the BM-WBCB.

1.2) Surface morphology and physical characteristics of adsorbent

Figure 2 displays the SEM image. As could be seen, the surface morphology of the BM-WBCB was primarily made up of hollow porous structures, which were influenced by the original structure of bamboo chopsticks. Upon closer examination, the surface morphology of BM-WBCB exhibits some periodic microstructures with a rough and deformed appearance. This distinct morphological outcome produced a large surface area.

To ascertain the specific surface area, pore diameter, and total pore volume of BM-WBCB, an examination of its physical characteristics was conducted. The result revealed a BET surface area, total pore volume, and pore diameter at 273.11 $m^2 g^{-1}$, 0.17 $cc\ g^{-1}$, and 2.54 nm, respectively.

1.3) Point of zero charge

The pH_{pzc} is essential in studying adsorption processes. It shows how functional groups on the adsorbent's surface affect the process. The pH_{pzc} is the pH at which the adsorbent's surface charge is neutral. The surface gets a positive charge if the pH_{pzc} is higher than the solution pH. If it's lower, the surface receives a negative charge. This study found that BM-WBCB had a pH_{pzc} of approximately 8.7 (Figure 3). When the pH was higher than 8.7, the surface had a negative charge, which effectively helped the BM-WBCB adsorb the cationic MB dye.

2) MB removal efficiency under various reaction conditions

2.1) Initial pH of the solution

The initial pH of the solution plays a crucial role in the adsorption process, impacting both the surface charge of the adsorbent and the chemical behavior of the adsorbate. Experimental data in Figure 4a illustrates the efficiency of MB adsorption under various pH solutions. The results demonstrate a positive correlation between pH and both the removal efficiency and adsorption capacity of biochar for MB. This tendency matched the findings of earlier research [17–20]. At acidic pH levels within the range of 4 to 5, the removal efficiency of MB is observed to be relatively low, approximately 60%. Conversely, the MB removal efficiency increased sharply as the pH increased to neutral. Adsorption efficiency of over 90% was observed in the strongly alkaline pH range (pH 10 to 12). These outcomes can be explained by the negatively charged surface of the BM-WBCB when the solution pH is approximately 8.7 ($pH_{solution} > pH_{pzc}$). The negative charge on the adsorbent surface attracts the positively charged MB molecules, leading to the observed adsorption phenomenon driven by electrostatic forces. In this experiment, pH 10 was selected as the optimal pH for MB adsorption on BM-WBCB. The removal efficiency reached 90.2% at this pH value, accompanied by an adsorption capacity of 4.5 mg g^{-1}.

2.2) Contact time

Figure 4b shows the behavior of MB removal efficiency and adsorption capacity of BM-WBCB over time. Within the first 05 minutes, there was a rapid increase in both MB removal efficiency and adsorption capacity, followed by a subsequent stabilization phase. The initial rapid adsorption can be attributed to a physical mechanism involving the transfer of mass between the solid and liquid phases [21–22]. The second stabilization phase of the adsorption process indicates that physical adsorption has reached equilibrium, and the system is experiencing minimal desorption of MB dye. This study determined that the required contact time for adsorption to reach equilibrium is 60 min. At this point, the MB adsorption efficiency of biochar was 94.2±2.6%, with an adsorption capacity of 4.7±0.1 mg g^{-1}.

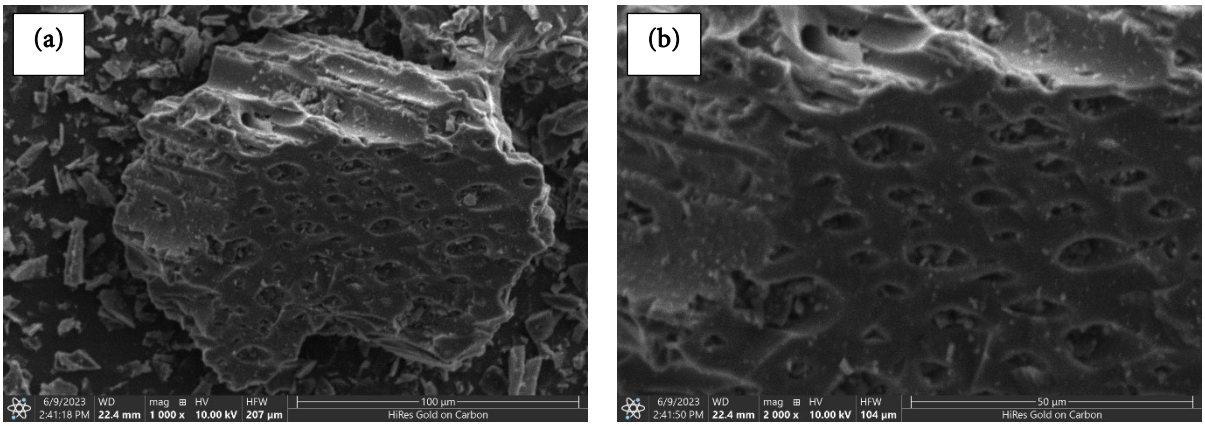

Figure 2 SEM image of the BM-WBCB: (a) 1000x magnification and (b) 2000x magnification.

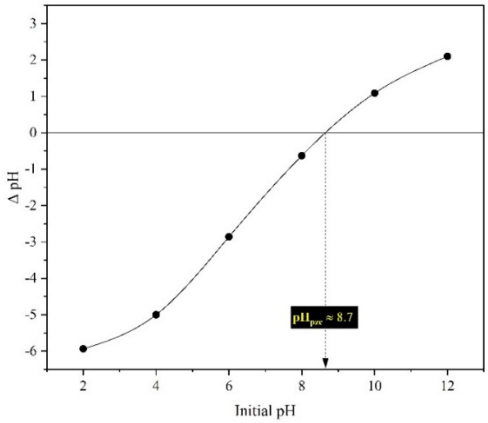

Figure 3 pH_{pzc} of the BM-WBCB.

2.3) Dose of BM-WBCB

The adsorption performance of MB dye on BM-WBCB was investigated at different dosages. Figure 4c shows that the removal efficiency of MB dye increased rapidly from 75% to 96% when the BM-WBCB dosage was increased from 0.1 g to 0.3 g (per 50 mL of MB solution). This suggests that the BM-WBCB dosage is a significant factor in the removal of MB dye. This increase in adsorption efficiency can be attributed to the higher number of adsorption sites available on the adsorbent as the dosage increases [23]. When the BM-WBCB dosage was further increased from 0.3 g to 0.5 g, the removal efficiency remained relatively stable. This suggests that the biochar layers may have overlapped and concealed some active adsorption sites at higher dosages.

Previous studies have shown that as the amount of adsorbent increases, the adsorption capacity decreases while the MB removal efficiency increases [16, 24]. In this study, a BM-WBCB dosage of 0.3 g was optimal for cost savings. At this dosage, the MB dye removal efficiency and adsorption capacity of BM-WBCB were 96.4% and 4.8 mg g^{-1}, respectively.

2.4) MB concentration

The effect of MB concentration (viz., 10, 20, 30, 40, and 50 mg L^{-1}) on adsorption performance was investigated in this study. As shown in Figure 4d, the MB removal efficiency was approximately 100% when the initial concentration was increased from 10 to 20 mg L^{-1}. However, when the MB concentration increased from 20 to 50 mg L^{-1}, removal efficiency decreased from 99.8 to 83.8%. The observed phenomenon can be explained by the following: as the MB concentration increases, the active sites on the adsorbent become saturated, leading to a decrease in the adsorption capacity. However, the MB removal efficiency still increases because the adsorbent can still remove MB from the solution, albeit slower. When the adsorbent dosage is fixed, the availability of active sites for the adsorption process becomes limited. Therefore, using a lower concentration of the MB solution ensures that the number of active sites is adequate for effectively eliminating all dye molecules [24].

In contrast, the adsorption capacity increased sharply (from 1 to 4.2 mg g^{-1}) when the MB concentration increased from 10 to 50 mg L^{-1}. The enhanced interaction between MB molecules and biochar particles could explain the increased adsorption capacity with increasing initial MB concentration. As the MB concentration increases, more MB molecules are available to interact with the biochar particles, leading to more MB molecules being adsorbed [25]. This trend is also consistent with previous studies on dye adsorption by biochar [16, 26–27]. Increasing the MB concentration generally increased the adsorption capacity but decreased the removal efficiency. This is because as the MB concentration increases, more MB molecules are available to interact with the adsorbent, leading to more MB molecules being adsorbed. However, the adsorbent can only remove a certain percentage of the MB molecules in the solution, so the removal efficiency decreases as the MB concentration increases. This study's optimal initial MB concentration was 30 mg L^{-1}, at which the removal efficiency and adsorption capacity reached 94.9% and 2.8 mg g^{-1}, respectively.

3) Adsorption isotherms

When an adsorbate and an adsorbent come into contact long enough, an adsorption isotherm is established. The interface and adsorbate concentrations in the bulk solution are dynamically balanced [28]. Typically, the adsorption isotherm is determined by varying the initial solute concentrations or the adsorbent dosage while keeping other conditions constant [29]. The selection of the Langmuir and Freundlich isotherm models in this investigation was based on their straight-forward parameterization. The graphical representation in Figure 5 exhibits the Langmuir and Freundlich plots concerning the adsorption of MB dye. As depicted in Figure 5a, the Langmuir model more accurately represents the adsorption process on BM-WBCB compared to the Freundlich model, as its values closely align with the experimental observations. Conversely, the Freundlich model (Figure 5b) displays a more significant deviation from practical matters, rendering it unsuitable for this study. The parameters and correlation coefficients for the Langmuir and Freundlich isotherm models are presented in Table 1. The higher correlation coefficient for the Langmuir model indicates that it is a better fit for the data, suggesting that the adsorption process under investigation conforms more closely to the Langmuir model. Consequently, monolayer adsorption is indicated, whereby the adsorption sites are identical and possess equivalent energetic characteristics.

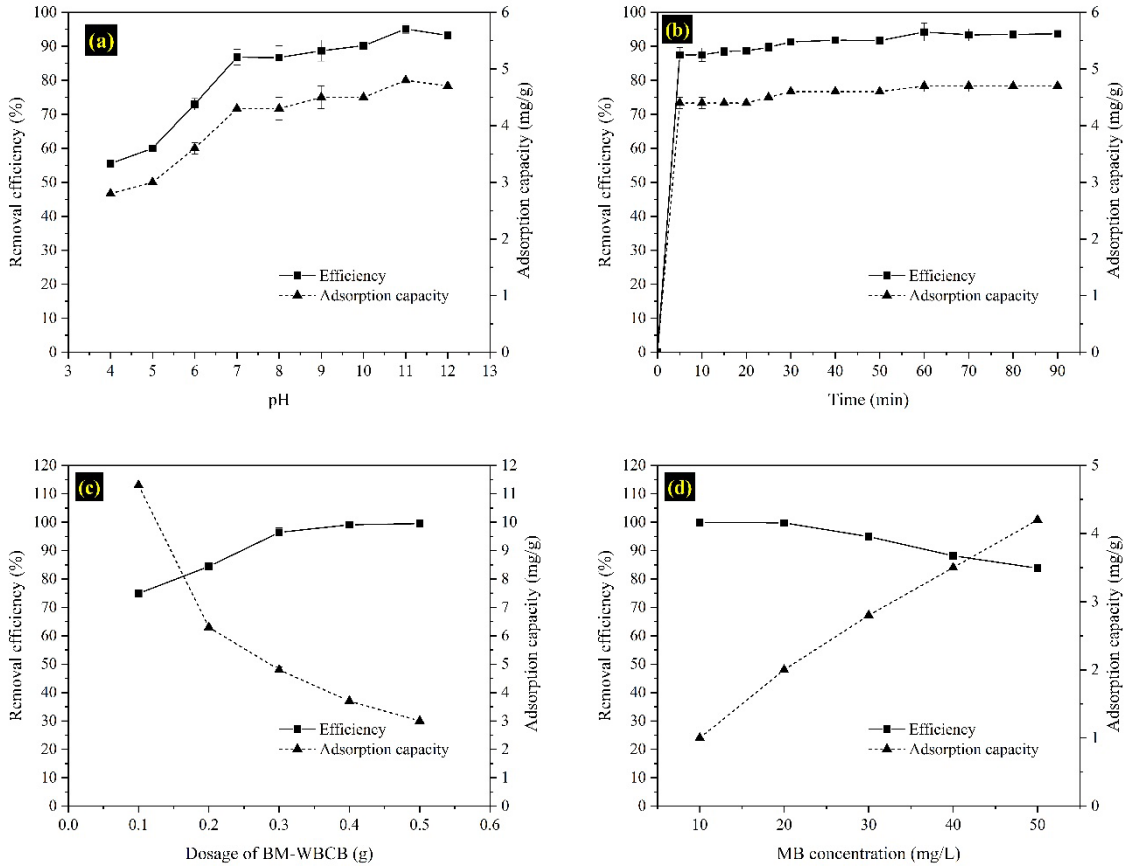

Figure 4 Effects of various experimental conditions on the adsorption of MB dye: (a) pH solution, (b) Contact time, (c) Dosage of BM-WBCB, and (d) MB concentration.

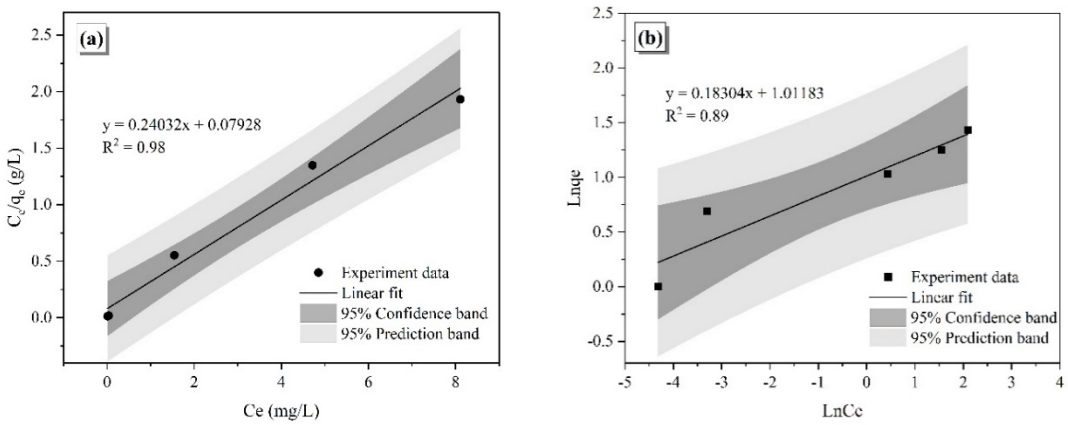

Figure 5 Adsorption isotherm models: (a) Langmuir and (b) Freundlich.

Table 1 The parameters of the Langmuir and Freundlich isotherm models

Isotherm models	Parameters value	R^2
Langmuir	Q_m = 4.2 (mg g^{-1})	0.98
	K_L = 3.03 (L mg^{-1})	
Freundlich	n = 5.5	0.89
	K_F = 2.8 (mg g^{-1})(L $mg^{-1})^{1/n}$	

4) Adsorption kinetic

This study investigated the kinetics and mechanism of the MB adsorption process by fitting the data into two kinetic models: the PFO model and the PSO model. The results presented in Figure 6 and Table 2 demonstrate that both models exhibit a solid fit for the experimental data, characterized by high correlation coefficients. However, comparing the R^2 values reveals that the PSO kinetics model (0.995) outperforms the PFO kinetics model (0.991). Consequently, the PSO model provides a more accurate description of the adsorption process of BM-WBCB. The PFO model is only applicable for characterizing the kinetics of the MB adsorption process during the initial stages when the adsorbent is still relatively free of MB molecules. As the adsorption process continues, the adsorbent becomes saturated with MB molecules, and the PFO model no longer provides an excellent fit to the data.

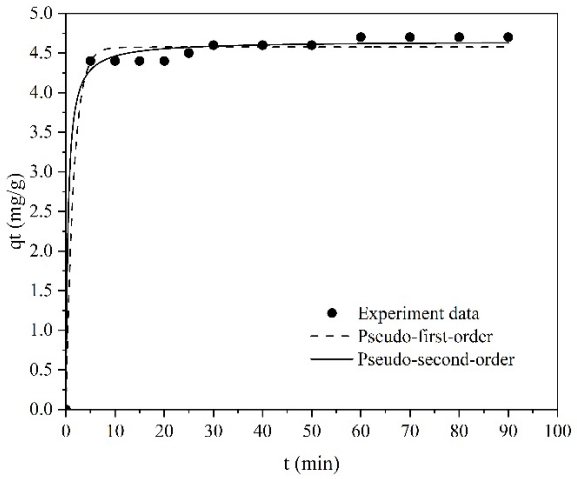

Figure 6 Adsorption kinetic models.

Table 2 The kinetic parameters for MB adsorption by BM-WBCB

Kinetic models	Parameters value	R^2
Pseudo-first-order	$q_{e,exp}$ = 4.7 (mg g^{-1})	0.991
	$q_{e,cal}$ = 4.57 (mg g^{-1})	
	k_1 = 0.63 (min^{-1})	
Pseudo-second-order	$q_{e,exp}$ = 4.7 (mg g^{-1})	0.995
	$q_{e,cal}$ = 4.65 (mg g^{-1})	
	k_2 = 0.5 (g mg^{-1} min^{-1})	

Conclusion

The slow-temperature pyrolysis process and ball-milling yielded biochar from waste bamboo chopsticks (BM-WBCB) characterized by a significantly increased specific surface area and a more intricate pore structure, indicative of its considerable adsorption potential. Various factors affecting the adsorption of methylene blue (MB), such as the pH, contact time, adsorbent dosage, and MB concentration, were found to influence significantly. The experimental data fit well with the Langmuir isotherm, with a maximum adsorption capacity of 4.2 mg g^{-1}. The attainment of equilibrium was observed within 60 minutes, and the adsorption kinetics conformed to the PSO model. Overall, this study has successfully demonstrated the potential of BM-WBCB as a viable adsorbent for the decolorization of dye-containing wastewater. By utilizing waste bamboo chopsticks as a precursor material for biochar synthesis, the study achieved two significant outcomes: (1) the reduction of waste generation and (2) the production of a value-added adsorbent material. This approach aligns with the principles of circular economy and sustainable waste management.

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