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Adsorptive Removal of Crystal Violet from Aqueous Solution Using Banana Pseudo Stem Biochar: Batch Adsorption Study

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Abstract

Synthetic dyes, such as crystal violet (CV), are extensively employed in many industries and contribute major environmental risks when released into water bodies. Agricultural waste biochar has been investigated for dye removal; however, studies on banana pseudo stem (BPS) biochar are limited. In particular, studies using BPS biochar produced at higher pyrolysis temperatures are scarce. Therefore, this study explores the adsorptive removal of BPS biochar produced through slow pyrolysis at 500°C for 1 h, a temperature selected to enhance the surface area and porosity for the removal efficiency of CV from aqueous solution. The effects of operating adsorption parameters, such as solution pH, biochar dosage, initial dye concentration, contact time and temperature, were assessed by batch adsorption experiments. The maximum CV percent removal of 96.9% with 4.85 mg/g of adsorption capacity was achieved at optimal conditions of pH 6, 0.4 g of biochar dosage, an initial CV concentration of 20 mg/L, 90 min of contact time, and at the temperature of 35°C. For the adsorption isotherm study, the experimental results were better fitted to the Langmuir model with a maximum adsorption capacity of 23.09 mg/g. Meanwhile, the kinetic study was found to be best fitted to the pseudo-second-order model. BPS biochar prepared at elevated pyrolysis temperature is a sustainable and efficient alternative to conventional adsorbents. The remarkable removal efficiency under mild conditions, combined with using agricultural waste as adsorbent feedstock, demonstrates the BPS biochar for practical use in dye-contaminated wastewater treatment.

1. Introduction

Crystal violet (CV), a synthetic triphenylmethane dye, named according to the IUPAC system as N [4-[bis (4-dimethyl-amino)-phenyl]-methylene]-2,5-cyclohexadiene-1-ylidine]-N-methyl-methenaminechloride. The chemical formula is $C_{25}H_{30}ClN_3$, with a molar mass of 407.99 g/mol. CV is extensively used in textile, printing, paper, and biological staining industries due to its vibrant colour and strong affinity for materials. However, its extensive utilisation has resulted in serious environmental issues. Untreated discharge of CV into aquatic environments can cause long-term damage to aquatic life by lowering light penetration and interfering with photosynthetic activity [1]. CV can also bioaccumulate in the food chain and affect biological activities, posing major human health hazards, including mutagenic and carcinogenic effects [2,3]. Therefore, eliminating crystal violet from wastewater is essential to protect both human health and the environment.

Recently, various techniques have been applied for dye removal, including chemical oxidation, coagulation-flocculation, membrane filtration, and biological treatments. Adsorption has attracted significant interest among these technologies because of its simplicity, high efficiency, low energy need, and ability to remove even low quantities of dyes [4]. Activated carbon is the most commonly used adsorbent owing to its high surface area and excellent adsorption capacity. However, its application is often limited by high production costs, regeneration difficulties, and the generation of secondary waste [5]. Biochar derived from agricultural waste has gained interest in current research as an eco-friendly alternative and is cheaper than activated carbon. Studies on coconut shells [6,7], sugarcane bagasse [8,9], rice husk [10,11], corn cob [12] and palm kernel shells [13,14] demonstrated that these wastes have been successfully converted into biochar for dye adsorption like methylene blue, crystal violet, malachite green and congo red and other dyes.

Banana production in Malaysia is significant, reaching nearly 312,968 tonnes in 2021 [15]. Of the total banana plants, the pseudo stem accounts for approximately 60% of the biomass, which is usually discarded as waste [16]. The banana pseudo stem is rich in cellulose, hemicellulose, and lignin, making it an appropriate precursor for biochar production [17]. Previous studies have shown the effectiveness of banana pseudo stem biochar in removing heavy metals such as copper [18], lead, and cadmium [19], as well as methylene blue dye [20,21] from aqueous solutions. These pollutants interact with the biochar surface via different adsorption mechanisms. For example, heavy metals usually rely on ion exchange and electrostatic attraction [18], while methylene blue adsorbs via electrostatic interactions and π-π stacking [21]. In comparison, CV is more difficult to remove due to its larger molecular size, which can cause blockage in small pores, and its strong tendency to bind to hydrophobic and aromatic surfaces. Therefore, for CV removal, the biochar must have a well-developed pore structure, high surface area, and sufficient aromaticity to ensure effective adsorption.

Research on using banana pseudo stem biochar for dye removal, particularly CV, remains scarce. To date, only [22] have examined biochar produced at a relatively low pyrolysis temperature, which typically results in lower surface area and less developed pore structure. Thus, this study aims to explore the potential of banana pseudo stem biochar produced at a medium pyrolysis temperature (500°C), a condition known to enhance surface characteristics for removing crystal violet from aqueous solutions. The effects of the adsorption parameters and the optimal conditions for maximum dye removal were assessed under batch adsorption conditions.

2. Materials and Methods

2.1 Chemical and Instrumentation

An analytical grade of CV powder (≥99% purity, Bendosen, Malaysia) was used in the preparation of the synthetic dye solution. A stock solution of 1000 mg/L of CV solution was prepared by dissolving 1.0 g of CV in 1 L of distilled water. The stock was diluted to the required concentration for further use in the standard calibration curve preparation. Meanwhile, for the adsorption experiments, the working solutions of CV (20 – 150 mg/L) were subsequently prepared from the stock solution by serial dilution using distilled water. Reagents including sodium hydroxide (NaOH) (≥98% purity, R&M Chemicals, Malaysia) and hydrochloric acid (HCl) (37%, R&M Chemicals, Malaysia) were used to adjust the pH value for the CV solution.

The following instruments were utilised throughout the biochar preparation and adsorption experiments. A digital balance (Sartorius BSA2235-CW, China) was employed for precise weighing. An oven (Venticell 55, Germany) was used to dry the raw banana pseudo stem, while a muffle furnace (Protherm PLF 110/5, Protherm Furnace, Turkey) was used to produce the biochar through slow pyrolysis. The solution pH was measured using a pH meter (Sartorius PB-10, China). For temperature-controlled adsorption studies, an incubator shaker (Protech SI-100D, Malaysia) was employed, with agitation set at 120 rpm to ensure uniform mixing and temperature control.

2.2 Preparation of Banana Pseudo Stem Biochar

Agriculture waste biomass of the middle section of the banana pseudo stems was collected from Ijok, Selangor. The stems were washed with tap water to remove impurities, cut into small pieces, and then air dried for five days, followed by oven drying for 24 h at 105°C [14]. The procedures continued with the preparation of biochar via the slow pyrolysis technique. The oven-dried stems were pyrolysed in a furnace at 500°C for 1 h. The produced biochar was ground, sieved for homogeneity, and finally stored in an air-tight container for subsequent use. Fig. 1 depicts the cleaned banana pseudo stem and the biochar produced.

Fig. 1 *Banana pseudo stem (a) Cleaned and cut into small pieces; (b) Biochar produced*

2.3 Batch Adsorption Procedure

The adsorption of crystal violet (CV) onto banana pseudo stem (BPS) biochar was studied through batch experiments under varying conditions: solution pH (2–10), biochar dosage (0.1–0.6 g), initial CV concentrations (20–150 mg/L), contact time (10–150 min), and temperature (25–65°C). To optimize these parameters, the one-factor-at-a-time (OFAT) method was selected due to its simplicity and practicality in the investigations. Through this approach, the effect of a specific factor on CV adsorption onto BPS biochar was identified by changing one parameter while the others remained constant. In each experiment, a specific amount of biochar was added to 100 mL of CV solution in a 100 mL Erlenmeyer flask. The mixtures were agitated at 120 rpm for designated contact times. After agitation, the solutions were filtered using Whatman No. 42 filter paper, and the residual CV concentration was determined using a UV-Vis spectrophotometer (Genesys 20 4001/4, Thermo Fisher Scientific, California) at 583 nm [22]. The CV percent removal ($R\%$) and the adsorption capacity (q_e) were calculated according to Equations (1) and (2),

$$R\% = \frac{(C_0 - C_e)}{C_0} \times 100 \tag{1}$$

$$q_e = \frac{(C_0 - C_e)}{M} \times V \tag{2}$$

where, C_0 and C_e are the initial and final CV concentrations (mg/L), respectively. M is the mass of the biochar (g), and V is the volume (L).

To ensure data accuracy and reliability, all experiments were performed in triplicate, and the results were reported as mean values accompanied by standard deviations for subsequent analysis.

3. Results and Discussion

3.1 Effect of Solution pH

The effect of solution pH on the CV removal and adsorption performance of BPS biochar is illustrated in Fig. 2. The lowest percent removal of 66.2% and adsorption capacity of 6.62 mg/g were observed at the initial condition of pH 2. Lower adsorption performance is caused by a high level of H^+ ions in acidic solutions, which compete with CV^+ dye molecules for accessible active sites [23]. The biochar surface becomes negatively charged as the pH of the CV solution increases, thus increasing the adsorption of the positively charged CV dye by electrostatic attraction. As the pH reached 6, both the percent removal and adsorption capacity of BPS biochar were significantly improved, achieving a maximum of 95.3% and 9.53 mg/g, respectively. Compared to a previous study using BPS biochar prepared at 300°C was only able to achieve the highest CV removal of 93.7% at pH 3 [22]. The enhancement is probably the result of the higher pyrolysis temperature, which causes the biochar surface to be less protonated, thus increasing the electrostatic interaction between the cationic dye molecules and the negatively charged sites. The removal efficiency and adsorption capacity were comparatively constant, nearly 94% and 9.4 mg/g when the pH increased from pH 7 to pH 10. This shows that surface charge stabilisation and electrostatic interactions take over the adsorption process after a specific pH threshold, which sustains a high removal rate. Kyi et al. [14] reported optimal CV removal at pH 7 using palm kernel shell biochar, attributing the enhanced adsorption to favourable electrostatic interactions under near-neutral conditions. Studies conducted by Amin et al. [24] on CV adsorption using *Eucalyptus camdulensis* tree wood biochar that was prepared at 800°C also confirm the effectiveness of BPS biochar under near-neutral pH conditions. Thus, the subsequent adsorption experiments were conducted at pH 6.

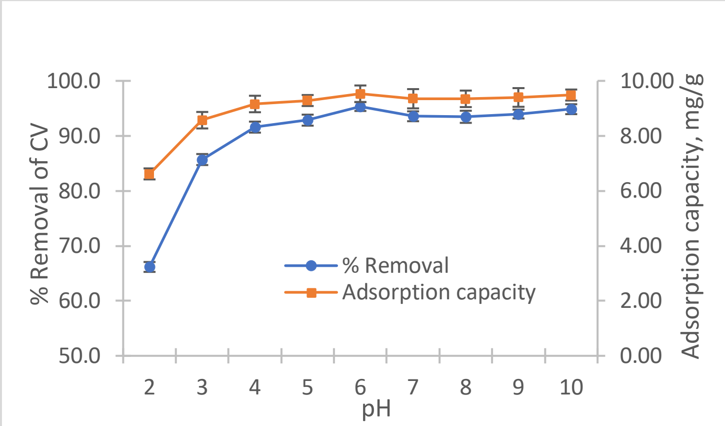

Fig. 2 *Effect of solution pH on the CV percent removal and adsorption capacity of BPS biochar (Dosage : 0.2 g in 100 mL, CV concentration : 20 mg/L, contact time : 60 min, temperature : 25 ℃). Values represent the mean. Error bars represent the standard deviation between three replicates (n=3)*

3.2 Effect of Biochar Dosage

Fig. 3 shows the effect of BPS biochar dosage on the removal efficiency and adsorption capacity of CV. It can be seen that the BPS biochar produced at 500°C showed a positive correlation between the dosage and percent removal of CV up to an optimal point. As the biochar dosage increased from 0.1 g to 0.4 g, the percent removal increased by 11.1% and reached the maximum removal of 97.4% with 0.4 g dosage. Higher dosage creates greater surface area and more vacant adsorption sites, thus facilitating more CV dye molecules to adsorb effectively [22]. Afterwards, increasing the dosage did not affect the percent CV removal. It was most likely due to the aggregation of the high concentration of adsorbent particles in the CV solution. Meanwhile, the adsorption capacity demonstrated a decreasing trend with increasing biochar dosage. The maximum adsorption capacity of 17.27 mg/g was recorded at 0.1 g dosage, and the value gradually reduced to 3.19 mg/g at 0.6 g. This can be attributed to the aggregation of the adsorbents due to the higher dosage, therefore decreasing the surface area of contact between the CV molecules and the adsorbent [25]. Moreover, the concentration gradient between the CV solution and the biochar surface diminishes with increasing dosages, which lowers the mass transfer driving force. The findings demonstrate that greater dosage enhances removal effectiveness, however, it may not be economical due to lower adsorption capacity per unit mass. An optimum dosage of 0.4 g balances a high percent removal and effective adsorption capacity for CV. Hence, further adsorption studies were performed using 0.4 g of biochar.

This behaviour is evident in biochars produced at higher pyrolysis temperatures (≥500°C) achieved higher adsorptive removal due to greater surface area, enhanced aromaticity, and well-developed porosity. Ghosh et al. [26] used coconut shell biochar pyrolysed at 600°C obtained >95% CV removal at just 0.2 g/100 mL dosage. *Gliricidia sepium* biochar produced at 700°C showed a higher adsorption capacity of 7.9 mg/g than lower-temperature biochars, highlighting the significant influence of pyrolysis temperature on adsorption efficiency [27].

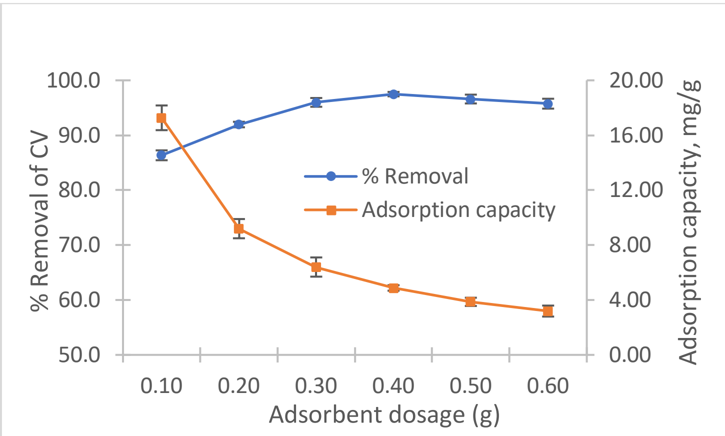

Fig. 3 *Effect of biochar dosage on the CV percent removal and adsorption capacity of BPS biochar (pH : 6, CV concentration : 20 mg/L, contact time : 60 min, temperature : 25 ℃). Values represent the mean. Error bars represent the standard deviation between three replicates (n=3)*

3.3 Effect of Initial Concentration

The results of the initial CV concentrations' effect on removal efficiency and the adsorption performance of BPS biochar are presented in Fig. 4. As the CV concentration increased from 20 to 150 mg/L, the percent removal decreased by 5.8%. The maximum removal of 97.5% was achieved with an initial CV concentration of 20 mg/L, which was attributed to huge amounts of active vacant sites on the biochar surface relative to the number of CV molecules, facilitating effective adsorption. The ratio of dye molecule to active site is ideal at this concentration, thus resulting in maximum surface coverage and saturation equilibrium. Further increasing the initial concentration causes the active sites to become saturated. This results in decreased removal efficiency, although the total dye uptake increases.

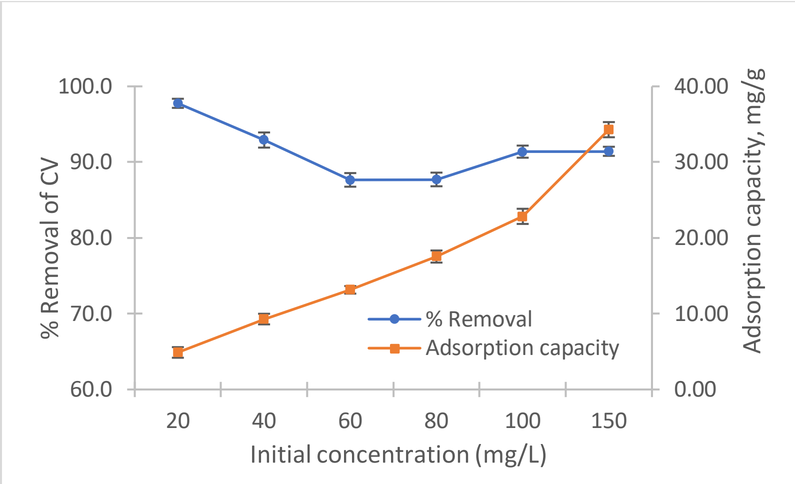

Fig. 4 *Effect of initial concentration on the CV percent removal and adsorption capacity of BPS biochar (pH : 6, dosage : 0.4 g in 100 mL, contact time : 60 min, temperature : 25 ℃). Values represent the mean. Error bars represent the standard deviation between three replicates (n=3)*

In contrast, the adsorption capacity increased significantly from 4.89 mg/g to 34.29 mg/g within the same range of CV concentration. This could be explained by the fact that higher dye concentrations are associated with elevated adsorption capacity values, suggesting the possibility of multilayer adsorption behaviour along with substantial dye-adsorbent interactions [28]. These findings are consistent with previous studies involving agricultural waste-derived biochar, confirming the capability of BPS biochar for treating wastewater with varying dye loads [14,24].

3.4 Effect of Contact Time

In the adsorption process, contact time is crucial as it defines the removal pathway and assesses the favourability of the pollutant removal procedure. Usually, the process starts rapidly before gradually slowing down until equilibrium is achieved. This theory is consistent with the findings of the contact time on the CV removal and adsorption performance of BPS biochar, as shown in Fig. 5. It can be seen that the percent removal of CV increased rapidly by 3.2% during the first 60 min, and continuously reached the highest removal of 96.2% when equilibrium was achieved at 90 min. In line with this, the adsorption capacity was similarly reached a maximum of 4.87 mg/g after 90 min. The initial rapid uptake is attributed to the abundance of available active sites on the biochar surface, which facilitated the binding of dye molecules. The subsequent 60 min of contact time indicate a consistent trend in percent removal and adsorption, proving that the equilibrium reaction was achieved. The saturation of active sites slowed the adsorption rate with time, confirming the hypothesis of site saturation. This behaviour is consistent with findings reported by previous studies, such as those by Jadhav and Thorat [23], who observed the adsorption of CV using biochar derived from agricultural waste. Sjahrir et al. [22] also utilised BPS biochar produced at 300°C, achieving maximum CV percent removal at 90 min of reaction, however, only 93.7%. This could be attributed to the lower surface area and pore volume developed for lower temperature biochars [23]. Later, the contact time of the following experiment was set at 90 min for optimal removal of CV.

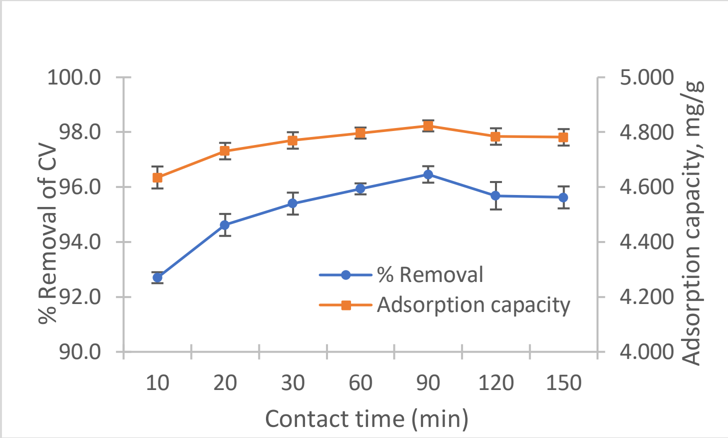

Fig. 5 *Effect of contact time on the CV percent removal and amount of CV adsorption capacity of BPS biochar (pH : 6, dosage : 0.4 g in 100 mL, initial concentration : 20 mg/L, temperature : 25 ℃). Values represent the mean. Error bars represent the standard deviation between three replicates (n=3)*

3.5 Effect of Temperature

In case of the effect of temperature, the CV removal efficiency and adsorption performance of BPS biochar were studied in the range of 25°C to 65°C. As depicted in Fig. 6, the highest percent removal was achieved at 35°C, with 96.9% and 4.85 mg/g of adsorption capacity. The percent removal and the adsorption capacity decreased slightly with increasing temperature over this optimal temperature, demonstrating an exothermic reaction of the adsorption process. Cheruiyot et al. [29] explained that this observation may be due to the weakening of binding forces, which facilitates the detachment of CV molecules from the biochar surface, resulting in reduced adsorption at higher temperatures. Besides that, higher temperatures may reduce the interaction time with active sites on the adsorbent and enhance the desorption process [30]. Thus, 35°C can be considered the optimal temperature for maximum adsorption efficiency and capacity in this study.

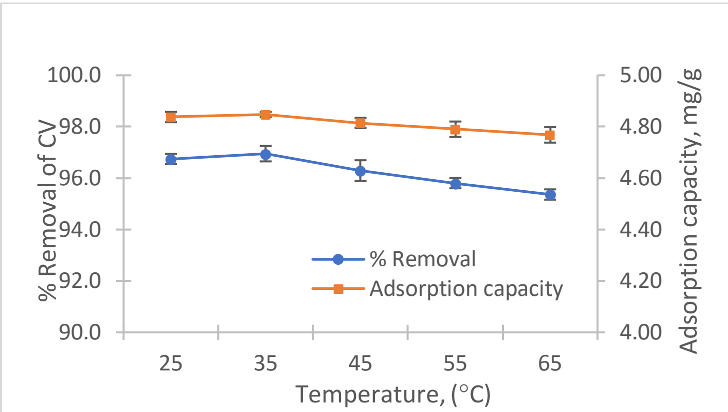

Fig. 6 *Effect of temperature on the CV percent removal and amount of CV adsorption capacity of BPS biochar (pH : 6, dosage : 0.4 g in 100 mL, initial concentration : 20 mg/L, contact time : 90 min). Values represent the mean. Error bars represent the standard deviation between three replicates (n=3)*

3.6 Adsorption Kinetics Studies

The adsorption kinetics mechanism of CV onto BPS biochar was investigated using pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models. The PFO model proposes that the adsorption process is influenced by the physical mechanisms, whereas the PSO model implies that chemical mechanisms influence the adsorption process. The kinetic experimental data were analysed using the linear equation of the PFO and PSO models as written below:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{3}$$

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$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

Where q_e and q_t are the adsorption capacity at equilibrium and at a given time (mg/g). k_1 (1/min) and k_2 (g/mg·min) are the PFO constant and PSO constant, respectively.

The fitting experimental kinetic data of the PFO and PSO models were compared, and the specific parameters are summarised in Table 1. According to the correlation coefficient (R^2), it is evident that the experimental data is best fitted to the PSO model. Besides that, the calculated adsorption capacity from the PSO model nearly matched the experimental adsorption capacity, 4.85 mg/g. These results suggest that the adsorption of CV onto the BPS biochar is primarily controlled by the chemisorption through electron sharing or exchange between the biochar and CV [14].

Table 1 *Kinetics adsorption parameters for CV adsorption onto BPS biochars*

Pseudo-First-Order			Pseudo-Second-Order		
R^2	q_e	k_1	R^2	q_e	k_2
0.324	0.115 mg/g	0.006 min^{-1}	0.999	4.796 mg/g	1.504 g/mg.min

3.7 Adsorption Isotherm Studies

The study employed the adsorption isotherm model to examine the interaction between the adsorbate and the adsorbent surface. Two isotherm models, Langmuir and Freundlich, were fitted to experimental data to determine the best-fitting isotherm model. Langmuir isotherm denotes monolayer adsorption on a homogeneous surface with limited and identical sites, while the Freundlich isotherm demonstrates the multilayer adsorption on a heterogeneous surface. The linear equation for Langmuir and Freundlich is represented in Equations (5) and (6), respectively.

$$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m} \tag{5}$$

$$\log q_e = \frac{1}{n}\log C_e + \log K_F \tag{6}$$

Where C_e is the CV concentration at equilibrium (mg/L); q_e is the adsorption capacity at equilibrium (mg/g); b is
the Langmuir isotherm constant (L/mg); q_m is the maximum adsorption capacity (mg/g); K_F is the Freundlich isotherm constant related to adsorption capacity (mg/g·L/mg$^{1/n}$); $\frac{1}{n}$ is the adsorption intensity. If $\frac{1}{n}$ = 1, the separation within the two phases is not dependent on the concentration. If the value of $\frac{1}{n}$ < 1, it shows normal adsorption. If $\frac{1}{n}$ > 1, it shows cooperative adsorption.

The separation factor (R_L) was used to assess the favourability of adsorption, which is calculated from the Langmuir isotherm, expressed as:

$$R_L = \frac{1}{1+bC_0} \tag{7}$$

Where, C_o is the initial CV concentration (mg/L). The values of R_L indicate the type of the isotherm to be either unfavourable (R_L > 1), favourable (0 < R_L < 1), irreversible (R_L= 0) or linear (R_L = 1).

From the linear regression of the Langmuir model, the obtained correlation coefficient (R^2) is 0.777, the maximum monolayer adsorption capacity (q_m) is 23.09 mg/g, and the Langmuir constant (b) is 0.341 L/mg. The separation factor (R_L) was calculated as 0.04, indicating highly favourable adsorption. For the linear regression of the Freundlich model, a significantly higher R^2 (0.902) was determined, showing a better fit of the experimental data compared to the Langmuir model. Given, the Freundlich constant (K_F) is 6.61 mg/g·L/mg$^{1/n}$. The value of $1/n$ is 0.4407, reflecting a favourable multilayer normal adsorption on a heterogeneous surface. Table 2 compares the adsorption capacity of the current study for CV to other previously published biochars.

Table 2 *Comparison of adsorption capacity of BPS biochar with other agricultural waste biochars for CV removal*

Biochar	Pyrolisis Temperature (°C)	Maximum Adsorption Capacity, q_m (mg/g)	Reference
Sugarcane bagasse	500	2.88	[9]
Palm kernel shell	350	24.45	[14]
Eucalyptus camdulensis tree wood	800	56	[24]
Gliricidia sepium tree wood	500	23.71	[27]
Palm petiole	700	209.00	[31]
Orange peel	800	203.81	[32]
Banana pseudo stem	500	23.09	This study

4. Conclusion

In this study, banana pseudo-stem biochar demonstrated significant potential for removing crystal violet from aqueous solutions under optimised conditions. Maximum percent removal was achieved at pH 6, where electrostatic interactions favoured CV adsorption. A dosage of 0.4 g provided the best balance between removal efficiency and adsorption capacity. The maximum CV percent removal of 96.9% and an adsorption capacity of 4.85 mg/g were attained under optimal adsorption conditions: 20 mg/L of initial concentration, 90 min of contact time, and at 35°C. Both removal and adsorption capacities slightly decreased with increasing temperature, indicating an exothermic reaction. Isotherm modelling showed that the experimental data fitted better to the Freundlich model, suggesting multilayer adsorption on a heterogeneous surface. The maximum adsorption capacity was 23.09 mg/g. Meanwhile, the kinetic study implies that the pseudo-second-order model best fitted the adsorption mechanism of CV onto BPS biochar. The results indicate that the BPS biochar demonstrated promising adsorption characteristics, with optimal conditions supporting its use in sustainable dye wastewater treatment. The regeneration and reusability of the BPS biochar for long-term application will be assessed for further investigation. Additionally, the performance of the biochar in real industrial wastewater will be evaluated to confirm its practical application for efficient wastewater treatment solutions.

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Conflict of Interest

Authors declare that there is no conflict of interest regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Noor Halini Baharim;* ***data collection:*** *Karteyaini Baskaran;* ***analysis and interpretation of results:*** *Karteyaini Baskaran, Noor Halini Baharim;* ***draft manuscript preparation:*** *Noor Halini Baharim;* ***revision of manuscript:*** *Noor Halini Baharim.*

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