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Original research article

Comparison of Biosorbent and Biochar Derived from Banana Pseudo Stem Waste for Crystal Violet Removal from Synthetic Wastewater

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ABSTRACT

Crystal violet (CV), a toxic carcinogenic dye commonly used for dyeing and colouring, is difficult to remove from effluents due to its complex structure. Currently, adsorption utilizing green adsorbent derived from abundant, low-cost agricultural wastes is an efficient and simpler technique than other dye removal methods. This study aims to evaluate the potential of banana pseudo stem (BPS) as biochar and biosorbent feedstock for CV dye removal. Batch experiments were conducted to investigate the effect of various adsorption parameters with one factor at a time (OFAT) analysis. The adsorbent characterization with the FTIR identified the presence of carboxylic, hydroxyl and amine groups, while SEM images showed rough, irregular pore structures, which resulted in dye molecules' strong adsorption onto the adsorbent surface. Using BPS biosorbent and BPS biochar, the highest CV removal of 91.9% and 93.7% was achieved at the same optimum adsorption conditions; pH 3, 2 g/L adsorbent dosage and 60 mg/L initial concentration. The adsorption on BPS biochar reached an earlier equilibrium time (90 min) as compared to the BPS biosorbent (110 min). The calculated maximum adsorption capacity, q_m, using the Langmuir model is 59.52 mg/g and 71.94 mg/g for BPS biosorbent and BPS biochar, respectively. Isotherm adsorption data for both adsorbents were better fitted to the Freundlich model. Therefore, the prepared BPS biochar has great potential as a promising adsorbent for removing CV dye from an aqueous solution.

Keywords: Adsorption; Crystal violet; Dye; Isotherm; Removal efficiency

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1. Introduction

Dye is employed widely in industries like colouring, paper, food and textile. The demand and consumption of synthetic dye in industries have expanded dramatically as compared to natural dyes due to its low cost of production and effective application. Crystal violet (CV) is one of the extensively cationic synthetic dyes used in dyeing and colouring purposes in industries such as clothing, cosmetics, pharmaceutical, textile, papers, leather and food processing. CV with the IUPAC name is N-[4-[bis[4-dimethylamino)-phenyl]-methylene]-2,5-cyclohexadien-1-ylidine]-N-methylmethanaminium chloride is also known as basic violet 3, gentian violet, and methyl violet 10B. The molecular structure of CV is given in Fig. 1, with the molecular formula of $C_{25}H_{30}N_3Cl$ and molecular weight of 407.98 g/mol.

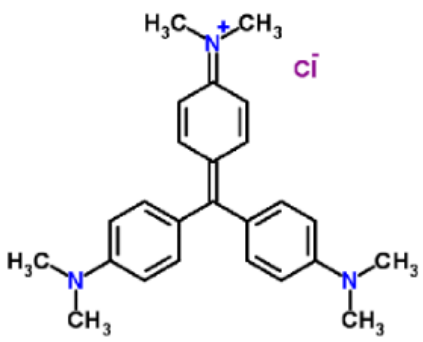

Fig. 1. Molecular structure of crystal violet.

Despite its aesthetic limitations, the presence of this purplish dye in the industrial effluent is connected to toxicological effects on aquatic species and the environment, subsequently creating significant threats to human health. CV dye can be absorbed through inhalation and ingestion and the skin, leading to horrible irritation, acute eye inflammation, breathing difficulty, nausea, hypertension, painful sensitization, and kidney failure [1]. The removal of these dyes from water and wastewater becomes an environmental challenge due to their synthetic origins and their mainly aromatic structures, which are extremely stable and biologically non-degradable. Their accumulation always leads to poor oxygenation of the water environment by preventing the photosynthesis of photosynthetic organisms [2].

Primary treatment methods used to remediate colour problems in wastewater may include filtration, filtration through a carbon filter, softening, reverse osmosis, chlorination and distillation. Most of these conventional methods are highly laborious, energy-consuming as well and high-cost. The adsorption process is one of the effective treatment methods, in the range of pollutants from moderate to low concentrations [3]. Conventional adsorbents such as activated carbon, zeolite, and silica gels have certain disadvantages including poor adsorption capacity, high cost and tendency to oxidize [4]. For this reason, attention has been given to natural properties like agricultural waste to be used as an alternative green adsorbent for the removal of dyes and colour pollutants. Adsorbents derived from coconut husk [5], rice bran [6], palm petiole [7], sugarcane bagasse [8] and corn stalk [9] have been applied in the study of removing CV dye from water. Considering it is renewable, employing adsorbent produced from agricultural waste can be both cost-effective and support environmental sustainability.

Banana pseudo stem (BPS), is a massive agricultural waste that is usually dumped in wet conditions or simply burned as waste without proper removal after fruit harvesting. The remaining waste cannot be left in the plantation field where high amounts of toxic gases such as carbon diox-

ide will be released if not managed properly thus leading to environmental problems. Due to its abundance and low-cost properties, BPS has been selected in this research as a precursor to producing two types of adsorbents, namely BPS biosorbent and BPS biochar for the removal of CV dye from synthetic wastewater.

2. Materials and Methods

2.1 Banana pseudo stem biosorbent and biochar preparation

Banana pseudo stem (BPS) was obtained from a village near to University of Selangor campus. The stem was washed with plenty of tap water and followed with distilled water to remove external dirt and impurities and cut into small sizes. The cleaned stem was air-dried overnight and continued with oven-dried (Venticell 55, USA) at 105°C for 24 h to remove moisture content [10]. Half of the dried material was mechanically ground, sieved to a particle size of 250 μm and denoted as BPS biosorbent.

The balance of the dried material was carbonized in a muffle furnace (PROTHERM, Turkey) for slow pyrolysis at 300°C for 1 h [11]. The produced biochar was ground, sieved into a size of 250 μm and labelled as BPS biochar. Both adsorbents were kept in different airtight containers for further use in the batch adsorption experiments.

2.2 Crystal violet solution preparation

An analytical grade of CV (Bendosen, Malaysia) was used in the experimental work. 1000 mg/L of CV stock solution was prepared by dissolving 1 g of CV powder with 1 L distilled water in 1 L of volumetric flask. The stock solution was diluted with an appropriate volume of distilled water to create experimental CV solutions with various concentrations. The pH of the CV solution was measured and adjusted to the required pH using 0.1 M HCl or 0.1 M NaOH.

2.3 Banana pseudo stem biosorbent and biochar characterization

The functional groups present in both adsorbents were identified by using an IR Tracer-100 FTIR (Shimadzu, Japan). The surface morphology of the BPS adsorbent was observed using scanning electron microscopy (SEM) (Zeiss supra 40, Germany) operated at 10 kV with 5.00 K magnification.

2.4 Batch adsorption experiments

Batch adsorption experiments were conducted at room temperature to investigate the effect of pH solution, adsorbent dosage, initial concentration and contact time on the CV removal efficiency using both BPS adsorbents. Experimental setup parameters that affect the adsorption have been optimized by one-factor-at-a-time (OFAT) technique. Each adsorption parameter experiment is detailed below.

2.4.1 pH solution experiment

The effect of pH solution on the removal efficiency of CV was investigated using 0.20 g of adsorbent in 10 different 250 mL conical flasks containing 100 mL of 50 mg/L CV solution. The pH solution for each flask was adjusted to a pH between 2 to 11. Using a rotary shaker, the flasks were agitated for 1 h at 120 rpm.

2.4.2 Adsorbent dosage experiment

Different adsorbent dosages (0.05 g, 0.10 g, 0.15 g, 0.20 g, 0.25 g and 0.30 g) were added into six 250 mL conical flasks containing 100 mL of 50 mg/L CV solution. The pH solution was adjusted to pH 3 (predetermined in 2.4.1). Using a rotary shaker, the flasks were agitated for 1 h at 120 rpm.

2.4.3 Initial concentration experiment

The effect of initial concentration on the removal efficiency of CV was investigated using six sets of 100 mL CV solution with different CV concentrations; 50, 60, 70, 80, 90 and 100 mg/L. The pH solution and adsorbent dosage used in this experiment were pre-determined in 2.4.1 and 2.4.2, respectively. The flasks were agitated for 1 h at 120 rpm using a rotary shaker.

2.4.4 Contact time experiment

The effect of contact time on the removal efficiency of CV was examined with different time intervals ranging from 10 min to 120 min. The pH solution, adsorbent dosage and initial CV concentration applied in this experiment were pre-determined in 2.4.1, 2.4.2 and 2.4.3, respectively. Using a rotary shaker, the flasks were agitated for 1 h at 120 rpm.

2.4.5 Adsorption isotherm experiment

This experiment was conducted using six conical flasks containing 50-100 mg/L of 100 mL CV solution. The pH solution was adjusted to pH 3 with 0.20 g of adsorbent dosage added into each flask and shaken for 1 h at 120 rpm using a rotary shaker. The equilibrium data were applied to the Langmuir and Freundlich isotherm models.

2.4.6 Adsorption analysis

The collected samples from the adsorption parameter experiment were then filtered and the residual CV dye concentration in the solution was analyzed using a UV-VIS Spectrophotometer (Hitachi, Japan) at the pre-optimized maximum wavelength of 583 nm. The removal efficiency was defined as percent removal (%R) and adsorption capacity (q_e) of CV using the BPS biosorbent and biochar was calculated according to Eqs. (2.1)(2.2):

$$\%R = \frac{(C_o - C_e)}{C_o} \times 100, \qquad (2.1)$$

$$q_e = \frac{(C_o - C_e)}{m} \times V, \qquad (2.2)$$

where, C_o and C_e are the initial and final CV concentrations (mg/L), respectively. V is the volume (L) and m is the mass of the adsorbent (g).

The experiments were carried out in triplicates for data accuracy, and the mean data and the standard deviation were presented. Statistical analysis was performed on the data using IBM SPSS Statistics 27 software, employing variance analysis (ANOVA) and Tukey's test for mean comparison at 95% reliability ($p < 0.05$).

3. Results and Discussion

3.1 Characterization of banana pseudo stem biosorbent and biochar

Fig. 2 shows the FTIR spectra of BPS biochar before and after adsorption, with a comparison to BPS biosorbent before adsorption. The analysis of the FTIR spectrum shows the presence of many peaks in the range of wavenumbers from 4000 to 500 cm^{-1}, which highlights the complex nature of the material analyzed. The BPS biochar had similar functional groups with the BPS biosorbent. The band at 3331.38 cm^{-1} (BPS biosorbent), 3336.93 cm^{-1} (BPS before adsorption) and 3339.61 cm^{-1} (BPS after adsorption) can be ascribed to the -OH stretching vibration of alcoholic or phenolic functional groups of the cellulosic and pectin molecule of BPS [12]. The band at 2918.96 cm^{-1}, 2908.45 cm^{-1} and 2920.22 cm^{-1} are due to aliphatic C-H stretching, which is present in polysaccharides [1]. For BPS biochar (before adsorption), the band at 1707.29 cm^{-1} represented the C=O

stretching. This might be caused by the pyrolysis, thus allowing the biochar to adsorb dye molecules [13]. For the BPS biosorbent, the band at 1597.87 cm^{-1} is notable for C=C stretching vibration. However, this band disappeared for BPS biochar (before adsorption) which could be due to the pyrolysis procedure [11]. This C=C vibration is noticed at 1587.33 cm^{-1} for BPS biochar after adsorption, proving the adsorption of CV onto BPS biochar. The appearance of C-N stretching at the bands around 1315 cm^{-1} and 1155 cm^{-1} is observed for biosorbent and biochar (before and after adsorption) [13]. Further evidence of interaction between BPS biochar (after adsorption) and the CV dye is provided by a drop in band height and a shift in this C-N stretching band position. The bands around 1030 cm^{-1} are linked to the C-O bond, which is present in cellulose and polysaccharides [12]. These bands of C-O are slightly deceased and widened after adsorption of the CV dye, indicating the presence of interactions between the CV and functional groups.

The SEM images of BPS biosorbent after adsorption, BPS biochar before and after adsorption are shown in Fig. 3. It can be observed that the external surface of the BPS biosorbent is very rough, while the BPS biochar surface is uneven, and irregular and contains heterogeneous pores, which enhance the adsorption of CV molecules onto the adsorbent surface. Mondal and Kar [14] and Yusuff [15] concluded similar observations using different adsorbents and dyes.

3.2 Batch adsorption analysis

3.2.1 Effect of pH of solution

Fig. 4 shows the effect of the pH of the solution on the percent removal of the CV solution investigated between pH 2-11. Both adsorbents achieved the highest removal at pH 3, with BPS biochar 0.6% higher than BPS biosorbent. This suggests that an acidic dye solution is more conducive to the CV removal from an aqueous solution. Previous studies by Sultana et al. [5] reported maximum CV dye removal was achieved at acidic conditions of pH 5 using coconut husk powder, while Goes et al. [16] and Amin et al. [17] found that the highest removal of CV using Carnauba straw and *Eucalyptus Camdulensis* biochar

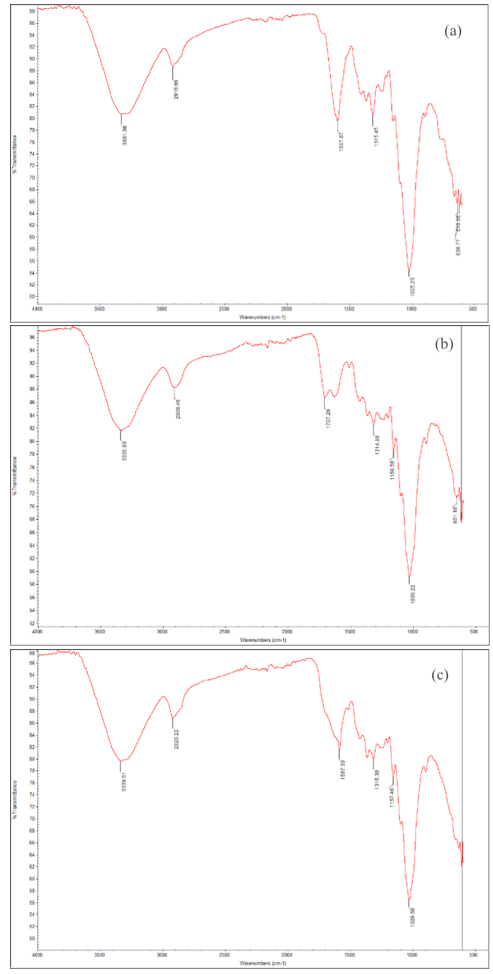

Fig. 2. FTIR spectra for BPS biochar, (a) before the adsorption; (b) after the adsorption of CV dye; (c) BPS biosorbent before adsorption.

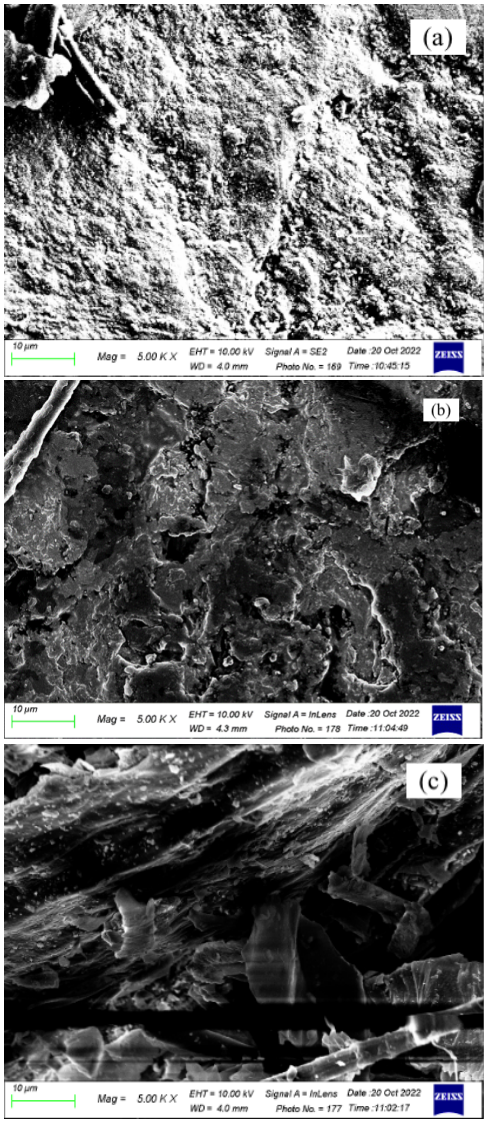

Fig. 3. SEM images for the surface of BPS, (a) biosorbent after adsorption; (b) biochar before adsorption; (c) biochar after adsorption.

was achieved at pH 4. The amount of positively charged CV dye and H^+ ions in the solution both drastically increased at a low pH value. Electrostatic attraction between the dye molecules and the adsorbent, facilitated by the negatively charged BPS surface, makes it easier to capture reactive and cationic dye on the adsorbent surface and increase adsorption. As the pH increased, the negative ions also increased in the solution. Rather than adsorbing to the adsorbent surface, the reaction between the cationic dye and the negative ions was expected to form a complex substance [5]. Hence, causing the adsorption removal of CV to be reduced at higher pH. Therefore, the following adsorption parameter experiments were set at pH 3 for both adsorbents.

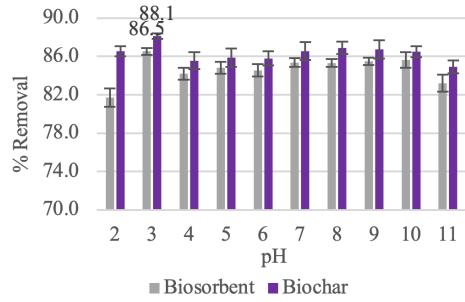

Fig. 4. Effects of pH solution on CV removal using BPS biosorbent and biochar.

3.2.2 Effect of adsorbent dosage

The effect of adsorbent dosage on the percent removal of the CV dye is shown in Fig. 5. Initially, the elevated trend in the removal of the CV was related to the availability of active sites for adsorption and additional adsorbent surface as the dosage increases [18]. With 2 g/L as the maximum removal adsorbent dosage, removal efficiency with BPS biochar was 2.2% higher than with BPS biosorbent. Afterwards, the percent removal decreased with the increasing dosage used in the experiment. This declining removal trend may be due to the unsaturated active site of the adsorbent during the adsorption process [19]. Besides that, more dosage caused the overlapping and aggregation of adsorbent, thus hindering the active site from being adsorbed by the adsorbate. As a result, the diffusion channel length increased and the surface area avail-

able for the adsorption of dye molecules reduced [20]. Furthermore, when the same dosage of adsorbent was used, BPS biochar often showed better removal efficiency than BPS biosorbent. Similar observations were also obtained by Liu et al. [11]. This might be due to the larger surface area and more heterogenous pores obtained for BPS biochar, thus resulting in higher percent removal of CV than BPS biosorbent. The adsorbent dosage was set to 2 g/L for both adsorbents in the following experiments.

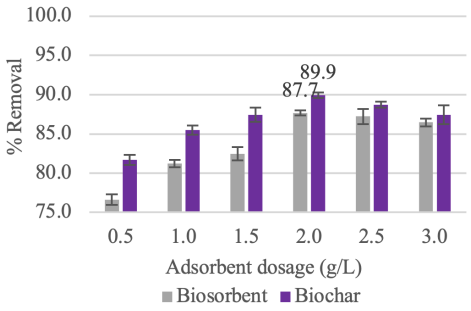

Fig. 5. Effects of adsorbent dosage on CV removal using BPS biosorbent and biochar.

3.2.3 Effect of initial concentration

The effect of the initial concentration of CV (50-100 mg/L) is illustrated in Fig. 6. Both adsorbents obtained the highest CV removal at 60 mg/L of initial concentration with 91.6% for biochar and 89.7% for biosorbent. CV removal achieved the highest percent at low initial concentrations because there is a low ratio of initial CV molecules to the vacant active sites on the adsorbent surface [19]. The following increase of CV concentration up to 100 mg/L caused the removal efficiency to decrease to 86.8% and 83.5% for BPS biochar and BPS biosorbent, respectively. This may be attributed to the impregnation of limited active sites on the adsorbent and increasing mass transfer caused by the increasing CV concentration [8]. Further experiments were carried out by using a 60 mg/L initial concentration of CV.

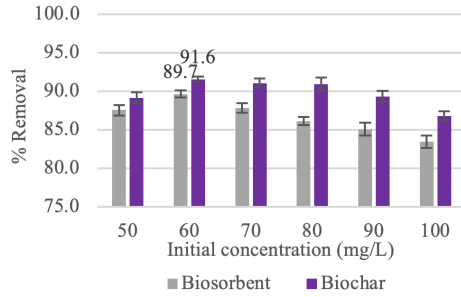

Fig. 6. Effects of initial concentration on CV removal using BPS biosorbent and biochar.

3.2.4 Effect of contact time

The adsorption process was also influenced by the contact time of the experiments. It can be seen in Fig. 7 that the percent CV removal was quite high at the beginning of the 30 min of reaction (especially with the biochar) due to the abundance of vacant sites available for the reaction. The percent removal continued to gradually increase until the adsorption equilibrium was attained after 90 min for biochar and 110 min for biosorbent. At this equilibrium time, the removal efficiency of 93.7% and 91.9% for BPS biochar and BPS biosorbent, respectively. Two steps of adsorption might be involved in this reaction. The first step was rapid surface adsorption followed by the intraparticle transport between the solution and porous surface of the BPS adsorbents. The results were consistent with previous studies on dye removal rates [21]. After achieving equilibrium, the percent CV removal was slightly decreased toward the end of experiments, as the active sites on the adsorbent surface were fully loaded with the dye molecules. Similar findings were reported by Sultana et al. [5] and Pathania et al. [18].

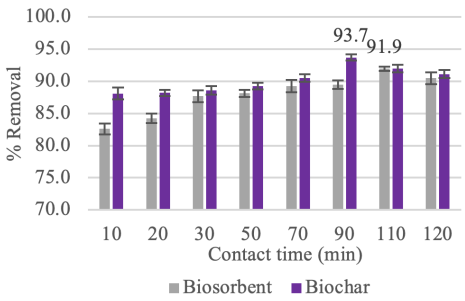

Fig. 7. Effects of contact time on CV removal using BPS biosorbent and biochar.

3.2.5 Adsorption isotherm

The adsorption molecules' distribution onto the biochar's solid phase at equilibrium conditions can be determined using adsorption isotherm studies. Analysis of the adsorption process was carried out using two common isotherm models, Langmuir and Freundlich.

Langmuir isotherm indicates the monolayer adsorption on a homogenized surface with a finite number of adsorption sites, whereas Freundlich isotherm describes the multilayer adsorption on a heterogeneous surface. The linearized equation for the models is tabulated in Table 1.

Table 1. Adsorption isotherm equations.

Models	Linearized equation
Langmuir isotherm	$\frac{C_e}{q_e} = \frac{1}{bq_m} + \frac{C_e}{q_m}$, (3.1)
Freundlich isotherm	$\log q_e = \frac{1}{n} \log C_e + \log K_F$,(3.2)

where q_e = adsorption capacity (mg/g), C_e = CV concentration at equilibrium (mg/L), b = Langmuir isotherm constant (L/mg), q_m = maximum adsorption capacity (mg/g), K_F = Freundlich isotherm constant related to adsorption capacity (mg/g), $\frac{1}{n}$ = 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 [22].

The separation factor, RL, for the prediction of the affinity between adsorbate and adsorbent is calculated from the Langmuir isotherm constant according to Eq. 3.3.

$$R_L = \frac{1}{1 + bC_o}, \qquad (3.3)$$

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 ($R_L = 1$).

According to R^2 in Table 2, it is evident that both BPS biosorbent and biochar adsorption isotherm were better fitted to the Freundlich model than the Langmuir model. The calculated maximum adsorption capacity, q_m, is 59.52 mg/g and 71.94 mg/g for BPS biosorbent and BPS biochar, respectively. The separation factor value, R_L indicates the favourable adsorption of CV onto both adsorbents at the applied experimental conditions. The adsorption of CV onto the biochar can be defined as normal and easy according to the $\frac{1}{n}$ value from the Freundlich model. The adsorption process of CV using live roots of *Eichhornia crassipes* and pine needle biochar was also described well by the Freundlich model [23,24]. A comparison of the maximum adsorption capacity of BPS biochar with other studied adsorbents is tabulated in Table 3.

4. Conclusion

This study shows that BPS biochar displays a better CV removal with a shorter equilibrium contact time than BPS biosorbent. The highest CV removal by BPS biochar was achieved at pH 3, using 2 g/L of dosage, 60 mg/L of CV concentration and 90 min of equilibrium contact time. The Freundlich model describes a better fit

Table 2. Adsorption isotherm parameters.

Isotherm model	Adsorbent	Parameters			
Langmuir	Biosorbent	R^2 = 0.881	b = 0.30 L/mg	q_m = 59.52 mg/g	R_L = 0.029
	Biochar	R^2 = 0.930	b = 0.10 L/mg	q_m = 71.94 mg/g	R_L = 0.035
Freundlich	Biosorbent	R^2 = 0.964	K_F = 16.7 mg/L	$\frac{1}{n}$ = 0.32	
	Biochar	R^2 = 0.936	K_F = 13.5 mg/L	$\frac{1}{n}$ = 0.49	

Table 3. Comparative study of maximum adsorption capacity, q_m, of various adsorbents for the CV removal.

Adsorbent	q_m (mg/g)	References
Palm kernel shell biochar	24.45	[25]
Eucalyptus Camdulensis biochar	54.64	[17]
Sugarcane bagasse biochar	99.5	[21]
Murraya koenigii stem biochar	35.71	[26]
Corncob modified with epichlorohydrin	71.43	[27]
Fennel seeds	13.47	[28]
Banana pseudo stem biochar	71.94	Current study

of the CV adsorption on both BPS adsorbents. The maximum adsorption capacity with BPS biochar and BPS biosorbent was 71.94 mg/g and 59.52 mg/g, respectively.

Finally, the comparison between BPS biosorbent and BPS biochar indicated that BPS biochar provides higher adsorption capacity for CV and is more efficient with a shorter time in removing this notorious dye from aqueous solution.

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