BATCH ADSORPTION OF CRYSTAL VIOLET ONTO ACID PRE-TREATED BANANA PSEUDO-STEM BIOCHAR: EFFECT OF OPERATIONAL PARAMETERS

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ABSTRACT

This study investigates the removal of crystal violet (CV) dye from aqueous solutions using biochar derived from locally grown banana pseudo-stem (BPS). The BPS was cut, air-dried, and then oven-dried at 105°C for 24 h, after which it was converted into biochar through slow pyrolysis at 300°C for 1 h. For pre-treated biochar (PT biochar), the dried BPS was soaked in 1 M H_2SO_4 for 24 h, rinsed to neutrality, and subsequently pyrolysed similarly. Batch adsorption experiments were conducted for both types of biochar to assess the effects of pH, adsorbent dosage, initial dye concentration and contact time. The untreated biochar achieved a maximum CV removal of 96.2% with adsorption capacities of 12.8 mg/g at a pH of 2, a dosage of 3 g/L, an initial concentration of 40 mg/L, and a contact time of 90 min. The PT biochar exhibited higher efficiency, with 98.1% removal and an adsorption capacity of 19.6 mg/g at pH 12, a dosage of 2 g/L, an initial concentration of 40 mg/L CV, and a contact time of 60 min. The utilisation of BPS presents a sustainable method for recycling agricultural waste into an eco-friendly, cost-effective, and efficient adsorbent for dye removal. The modification of biochar through acid pre-treatment significantly improves its adsorption performance, highlighting its potential for effective CV removal from water and wastewater, thereby contributing to enhanced water quality and environmental sustainability.

Keywords: *Agriculture waste, biochar modification, dye, pyrolysis, removal efficiency*

INTRODUCTION

Crystal violet (CV), also called gentian violet or basic violet 3, is a cationic triphenyl methane dye that is classified as a synthetic dye. The chemical formula of CV is $C_{25}N_3H_{30}Cl$, and the molar mass is 407.98 g/mol. The chemical structure of CV is depicted in Figure 1. CV is a basic dye that is 9% soluble in both alcohol and water (soluble 8.8%). This particular dye finds the most widespread application in the textile and paper manufacturing industries [1]. CV also has a wide range of applications, such as a biological stain, a dermatological agent, and an additive used in veterinary medicine to inhibit the formation of mould and intestinal parasites, as well as fungus, among other things [2]. However, due to the intricacy of their molecular structures, triphenyl methane dyes are notoriously difficult to eliminate from effluents [3]. It remains in various environments for an extended time, and microbes poorly metabolise it. Besides that, CV dye causes damage to the cornea and the conjunctiva that is permanent. This can cause slight discomfort to the eye as well as an uncomfortable sensitivity to light [4]. It also causes severe damage to mammalian cells and, if ingested in large quantities, can irritate both the digestive system and the skin [5]. The CV must be removed from wastewater before it is discharged to ensure the continued existence of both humans and other aquatic species [6].

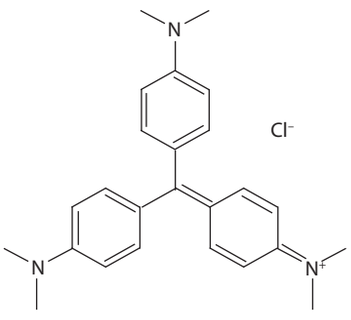

Figure 1 Chemical structure of crystal violet

The treatment of dye in an environment has traditionally involved the use of conventional treatment methods, which encompass physical, biological, and chemical approaches. Various physical processes, such as coagulation, biological treatment, oxidation, membrane filtration, and adsorption, are utilised in the treatment of dyes. Each of the treatment methods has a unique level of effectiveness, as well as some drawbacks. Particularly among the methods, adsorption is considered the most appealing due to its numerous benefits, including ease of use, low cost, effectiveness over a broad pH range, excellent performance, and potential for recycling the adsorbent through regeneration [7]. Activated carbon is a common adsorbent used in many applications due to its high surface area and excellent adsorption capacity. However, its

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application has been limited by the high price, resource-intensive and often inefficient for vat dyes. Its regeneration is complex and environmentally burdensome [8]. These limitations underscore the pressing need to develop alternative, low-cost, renewable adsorbents with improved selectivity and sustainability.

Biochar is a relatively new alternative adsorbent produced by heating biomass in the absence of oxygen, resulting in a highly porous material with a large surface area. Biochar can be produced from a variety of waste materials, including agricultural residues, forestry residues, and municipal solid waste, thereby reducing waste and providing a source of renewable energy. Agricultural wastes from plant resources, such as maize straw, sunflower straw, wheat straw, orange peels, and chestnut shells, were utilised to produce biochar for the removal of heavy metal ions, dyes, and organic pollutants from aqueous solutions [9]-[10]. However, compared to activated carbon, biochar may have lower adsorption capacity and selectivity for certain compounds, depending on the type of feedstock used, the production temperature and time, and the activation method [11]. Therefore, biochar needs to be modified to enhance its physicochemical adsorptive removal properties for specific adsorption applications and ensure better contaminant removal compared to unmodified biochar.

One common approach for biochar modification is acid activation. This can be performed by treating the biomass or biochar before or after the pyrolysis. Acid treatment cleans the surface, removes ash and creates more pores, which increases the surface area. Besides that, the procedure introduces functional groups like -OH and -COOH that would attract cationic dye like crystal violet. Previous studies have demonstrated that acid-modified biochar exhibits superior performance in dye removal compared to unmodified or untreated biochar [6]-[7].

Banana pseudo-stem is the major waste product of the banana plant, which grows one of Asia's most popular fruits. When bananas are harvested, the stems are usually cut down and left to decompose on the plantation, where they will eventually decompose into organic waste, potentially damaging the environment. Banana waste leftovers can be effectively recycled to create adsorbents for the remediation of polluted water. Due to their high lignin and low cellulose contents, the stems are a promising raw material for making biochar, which ideally has a high production yield, a sizable specific surface area (SSA), a porous structure, and a high fixed carbon content. Banana stem absorbent was used to remove heavy metals from wastewater, including Pb(II) [12], Zn(II), Mn(II), and Cu(II) [13], as well as cadmium [14]. Nearly 97% of crystal violet removal, with an adsorption capacity of 4.85 mg/g, was achieved using banana stem biochar produced at a medium pyrolysis temperature [15]. A study conducted by Rigueto et al. [16] using banana pseudo-stem biochar for removing methylene blue and safranin dye successfully achieved maximum adsorption capacities of 33.78 mg/g and 21.74 mg/g, respectively. So far, no work has demonstrated the use of pre-treated banana pseudo-stem biochar with acid modification for removing crystal violet. Therefore, this study investigates the potential of acid pre-treated banana pseudo-stem biochar to remove crystal violet from aqueous solutions. Through batch adsorption experiments, the effects of pH, adsorbent dosage, initial dye concentration, and contact time were investigated to determine the optimal adsorption conditions for achieving maximum crystal violet removal efficiency.

METHODOLOGY

Materials

The banana pseudo-stems (BPS) of *Musa Paradisiaca* were collected from a plantation in Bestari Jaya, Selangor. CV powder (≥ 99% purity, Bendosen, Malaysia), sodium hydroxide (NaOH) (≥ 98% purity, R&M Chemicals, Malaysia), sulphuric acid (H2SO4) (95.98%, R&M Chemicals, Malaysia) and hydrochloric acid (HCl) (37%, R&M Chemicals, Malaysia) were all of analytical grade. CV stock solution was prepared by dissolving 1.0 g of CV powder in 1000 mL of distilled water. The stock solution was diluted to the appropriate concentration for use in preparing the standard calibration curve and the batch adsorption experiments. The absorbance of the CV solution was measured at a wavelength of 583 nm using a UV-Vis spectrophotometer (Genesys 20 4001/4, Thermo Fisher Scientific, California) [15]. The pH of the CV solutions was adjusted by adding 0.1 M NaOH and 0.1 M HCl solution, and the pH was measured using a pH meter (Sartorius PB-10, China).

Preparation of Biochar and Pre-treated Biochar

The collected BPS were peeled back one layer at a time, cut into small pieces and rinsed well under running water to remove any traces of dirt. Then, the stems were air-dried for three days and subsequently oven-dried (Venticell 55, USA) at 105°C for 24 h. Figure 2 shows the oven-dried BPS.

The dried BPS was divided into two portions: one for the preparation of untreated biochar and the other for pre-treated biochar. The biochar was produced by a process called slow pyrolysis, which involves placing the dried biomass in a sample holder and heating it in a furnace (PROTHERM, Turkey) at 300°C for one hour in the absence of oxygen [17]. The produced biochar was ground, sieved, and kept in an airtight container. This untreated biochar was denoted as biochar in the discussion.

Figure 2 Banana pseudo-stems after being dried in an oven

To prepare the pre-treated biochar, 10.0 g of dried biomass was added to 50 mL of 1 M H_2SO_4 solution for 24 h. After the treatment, the biomass was filtered, washed with plenty of distilled water to neutrality and dried. The pre-treated samples were subjected to the same pyrolysis procedure, ground, sieved, and then stored in a container for further use. This acid pre-treated biochar was labelled as PT biochar.

Batch Adsorption Experiment

The effect of the operating adsorption parameters on the removal efficiency of CV using both untreated biochar and PT biochar was investigated in batch mode. The adsorption experiments were conducted by varying the solution pH (2 to 12), adsorbent dosage (1 g/L to 6 g/L), and initial CV concentration (10 mg/L to 60 mg/L) and contact time (10 min to 120 min). The adsorption parameter was optimised using the one-factor-at-a-time (OFAT) technique. In each experiment, a specific amount of biochar was added to 100 mL of CV solution in a 250 mL Erlenmeyer flask. The mixture was agitated at 120 rpm for predetermined contact times. After that, the mixture was subjected to further filtration and the absorbance of residual CV concentration was measured using a UV-Vis spectrophotometer. The concentration of CV was calculated based on the linear equation obtained from the calibration curve of the CV solutions, equated as:

$$y = 0.1285x + 0.0522 \quad (1)$$

The percentage of CV removal efficiency (*%R*) and adsorption capacity (q_e) were calculated as:

$$\%R = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

$$q_e = \frac{(C_0 - C_e)}{m} \times V \quad (3)$$

where C_0 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 unit of q_e is mg/g.

All batch experiments were repeated in triplicate to ensure the reliability and accuracy of the data. The results were presented as the mean concentrations with the standard deviations for the data analysis.

RESULTS AND DISCUSSION

Effect of Solution pH

The pH level is one of the most crucial and influential parameters in the adsorption process, as well as in the surface chemistry of the adsorbent. Different acidic and alkaline environmental conditions significantly influence the ionisation of functional groups on the adsorbent surface, thereby affecting the adsorption of dye molecules onto the adsorbent. The effects of solution pH on the percentage removal efficiency of CV are shown in Figure 3. The untreated biochar achieved its maximum percentage removal of 94.2% at pH 2, then decreased to nearly 4.0% until pH 8, which could be attributed to various surface functional groups that enhance hydrogen bonding and π-π interactions. Afterwards, the removal efficiency remains slightly increased by 2.0% and reaches 93.3% at pH 12, indicating the broad applications of the surface biochar. Therefore, this finding suggests that untreated biochar is more effective in acidic to neutral conditions.

In contrast to the PT biochar, the percentage removal of CV gradually improved from 71.6% at pH 2 to a maximum of 95.6% at pH 12. The lower removal percentage in acidic conditions is likely due to surface protonation and the low availability of functional groups resulting from acid pre-treatment [18]. The CV removal was then increased progressively with pH, which can be explained by the increased electrostatic attraction between the negatively charged biochar surface and the cationic dye at alkaline pH [19]. The acid pre-treatment improves the surface charge and increases the availability of active sites, thereby enhancing adsorption under alkaline conditions. This demonstrates that acid pre-treatment effectively modified the surface chemistry, leading to greater adsorption efficiency at higher pH values. It is suggested that PT biochar performs better in an alkaline environment, making it a

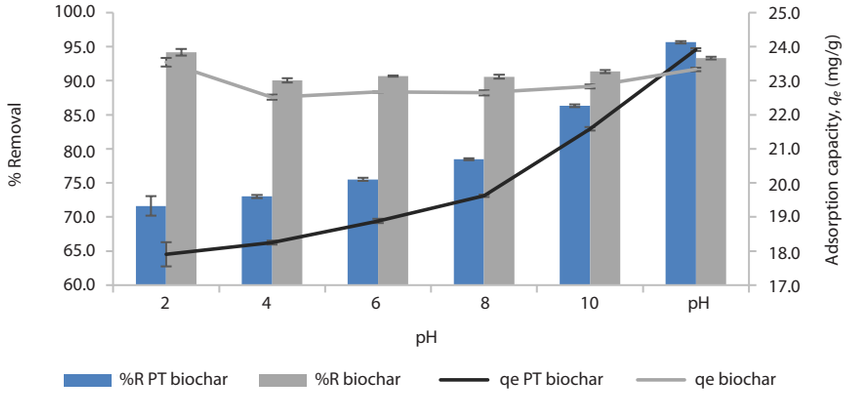

Figure 3 Effect of solution pH [Conditions: Dosage = 2 g/L; Concentration = 50 mg/L; Contact time = 60 min; Temperature = 25°C]

promising adsorbent for high-pH wastewater. Jawad et al. [20] reported similar findings, indicating that the increase improved the adsorption of cationic dyes onto negatively charged functional groups generated by acid pre-treatment through attractive electrostatic forces. Meanwhile, Xu et al. [21] highlighted an improvement in dye removal at an alkaline pH using modified corn husk biochar. Therefore, determining the optimal pH is vital for maximising the removal efficiency of CV using acid-modified biochar.

The solution pH strongly influenced the adsorption capacity of both untreated and PT biochar. For untreated biochar, the highest q_e (23.5 mg/g) was achieved under acidic conditions (pH 2), where the protonation of surface functional groups enhanced the interaction with CV molecules. However, the q_e gradually decreased as the pH increased towards neutral, indicating reduced affinity of the adsorbent surface for the dye. Meanwhile, the PT biochar showed lower q_e values in acidic medium but improved significantly with increasing pH, reaching its maximum of 23.9 mg/g at pH 12. This trend can be explained by the presence of negatively charged functional groups generated through acid activation, which strengthened the electrostatic attraction with the cationic dye under alkaline conditions. These results clearly indicate that untreated biochar exhibits higher adsorption in an acidic medium, while acid-pre-treated biochar performs better in alkaline conditions, highlighting the role of surface chemistry in determining adsorption efficiency.

Effect of Adsorbent Dosage

The effect of adsorbent dosage, ranging from 1 g/L to 6 g/L, on the CV adsorption was examined at optimum pH levels of 2 and 12 for untreated biochar and PT biochar, respectively. As shown in Figure 4, the removal efficiency of CV exhibited a positive correlation with increasing adsorbent dosages. For untreated biochar, the CV removal increased from 91.8% at a 1 g/L dosage to a maximum removal of 95.2% at a 3 g/L dosage. At higher dosages, the removal percentage gradually dropped to 93.3%, implying no significant CV adsorption on the biochar surface. Meanwhile, the PT biochar consistently achieved higher removal efficiency, starting from 93.0% at 1 g/L and reaching approximately 95.5% at 2–5 g/L, with only a slight decline of 0.4% at 6 g/L. The highest removal of 95.8% was achieved with a 2 g/L dosage. The enhanced performance of PT biochar can be attributed to acid pre-treatment, which likely increases the active surface area and porosity of the biochar, and introduces functional groups such as -OH and -COOH that favour CV adsorption. Additionally, the surface charge of biochar can be altered by acid treatment, thereby improving cationic dye adsorption under alkaline conditions [22].

An interesting, similar finding was observed for both biochars at higher dosages. The increasing amount of biochar reduces the availability of CV molecules relative to the number of active sites, leading to surface saturation and minimal improvement in removal efficiency. Furthermore, the total adsorption capacity may be limited by particle agglomeration at high doses, which may block active sites and decrease the effective surface area [23]. The same results were reported by AL-Shehri et al. [24] and Kyi et al. [3]. Therefore, the following experiments were conducted using the optimum dosages corresponding to the highest removal efficiencies: 3 g/L for untreated biochar and 2 g/L for PT biochar.

While removal efficiency marginally increased with increasing dosage, the adsorption capacity of both untreated and PT biochar declined. At lower dosages, both biochars showed higher q_e values due to greater availability of dye molecules relative to active sites. PT biochar achieved higher capacities (46.5 mg/g at 1 g/L dosage) compared to the untreated biochar (45.9 mg/g), confirming the beneficial effect of acid activation. However, as the dosage increased beyond the optimum levels of 3 g/L and 2 g/L for untreated and PT biochar, respectively, the adsorption capacities declined due to site saturation, particle agglomeration, and a reduced dye/adsorbent ratio.

Effect of Initial Concentration

The effect of the initial CV concentration on the percentage removal of CV is presented in Figure 5. Both biochars exhibited a similar trend, where the percentage removal increased from 10 mg/L to 30 mg/L, then reached the highest percentage at 40 mg/L, and

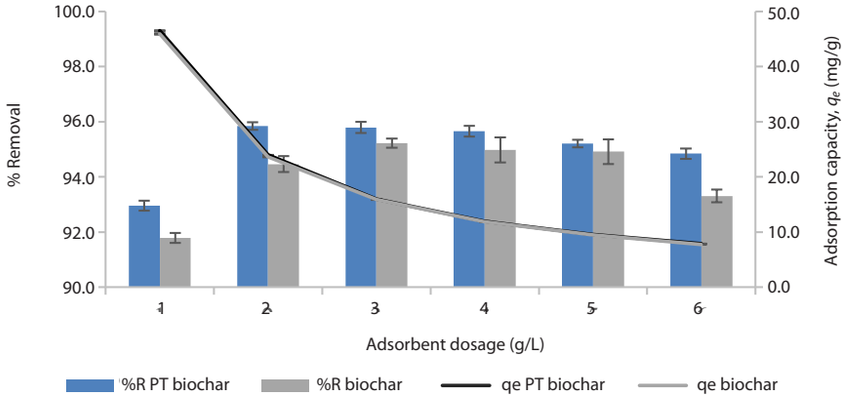

Figure 4 Effect of adsorbent dosage [Conditions: pH = 2 (Untreated biochar), 12 (PT biochar); Concentration = 50 mg/L; Contact time = 60 min; Temperature = 25°C]

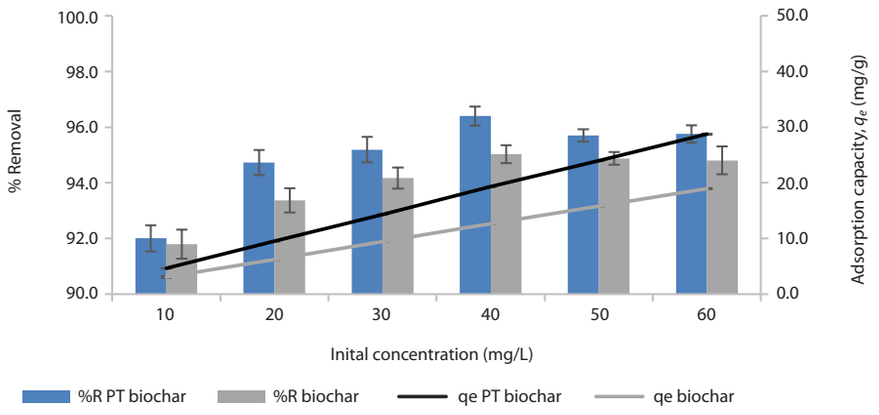

Figure 5 Effect of initial concentration [Conditions: pH = 2 (Untreated biochar), 12 (PT biochar); Dosage : 3 g/L (Untreated biochar, 2 g/L (PT biochar); Concentration = 50 mg/L; Contact time = 60 min; Temperature = 25°C]

continued to slightly decrease towards 60 mg/L. From 10 mg/L to 40 mg/L, the removal efficiency increased by 4.4% and 3.2% for PT biochar and untreated biochar, respectively. This improvement can be attributed to the increased availability of adsorption sites on the adsorbent surface at lower CV concentrations, thereby allowing more dye molecules to be adsorbed effectively. Above 40 mg/L, the removal efficiency was slightly reduced due to the saturation of the adsorption sites and the fact that CV molecules could no longer be taken up in the adsorption process [25]. Additionally, the limited number of active sites restricts the adsorption capacity at higher concentrations, which causes a plateau or slight decrease in percentage removal, even though the driving force for mass transfer may still be sufficient [17].

For PT biochar, the highest removal percentage was recorded at 96.4% with a concentration of 40 mg/L. Meanwhile, untreated biochar achieved a maximum removal of 95.0% at the same concentration, which was 1.4% lower than that of PT biochar. The enhanced performance of PT biochar can be attributed to the improved interactions between the dye molecule and the adsorbent, resulting from the enhanced pore structure and increased abundance of functional groups generated during acid pre-treatment [26]. This finding aligns with previous studies, which have shown that surface modification of biochar significantly improves the adsorption efficiency, particularly for cationic dyes [5],[27].

The adsorption capacity of both untreated and PT biochars increased with higher initial dye concentrations, reflecting greater driving force for mass transfer. Both biochars achieved their optimum capacity at 40 mg/L, with the untreated biochar reaching 12.7 mg/g, while the PT biochar achieved 19.3 mg/g, indicating the positive effect of surface modification. Further increases to 60 mg/L raised q_e values to 19.0 mg/g and 28.7 mg/g for untreated and PT biochar, respectively, although percentage removal slightly declined due to site saturation. These results indicate that higher initial concentrations enhance the quality of the effluent until equilibrium is reached, with acid-pre-treated biochar consistently demonstrating superior adsorption capacity compared to untreated biochar.

Effect of Contact Time

The contact time is another crucial factor in determining the optimum reaction time for the adsorption process, as it governs the interaction between the adsorbate and adsorbent surfaces. In this study, batch experiments were conducted with contact times ranging from 10 to 120 min, and the results are depicted in Figure 6. For the untreated biochar, the removal efficiency increased rapidly by approximately 9.0% within the first 20 min. Meanwhile, PT biochar exhibited a smaller increase of about 1.4% during the same period. Initially, the higher adsorption rate observed for both biochars is closely related to the abundant availability of active sites and large surface area at the beginning of the process, enabling rapid uptake of CV molecules from the solution [28].

After the initial phase, the percentage removal for both biochars steadily increased as the adsorption process continued, though at a slower rate due to the progressive saturation of active sites. The maximum CV removal using untreated biochar and PT biochar was achieved at 90 min (96.2%) and 60 min (98.1%), respectively. As shown in Figure 6, the PT biochar achieves equilibrium more quickly than the untreated biochar. This might be that acid pre-treatment significantly enhances adsorption kinetics by increasing surface functional groups and improving pore accessibility, which promotes stronger electrostatic attraction and faster dye uptake [29]. After reaching the maximum removal, the percentage removal was slightly decreased towards the end of the experiment. This may be due to desorption, intraparticle diffusion limitations, or the redistribution of dye molecules between the liquid phase and adsorbent surface [30]. Darama et al. [31] reported that biochar derived from prina achieved an optimum contact time of 60 min, with a removal efficiency of 99.1% higher than that of natural prina (75.4%). Bui et al. [26] have also confirmed that pre-treated biochar with acid can substantially reduce the time required to reach equilibrium while enhancing overall removal efficiency.

The adsorption capacity of both biochars was increased with contact time until equilibrium was reached. A rapid increase in q_e was observed at the initial time due to the abundant availability of active sites, enabling fast dye adsorption. The untreated biochar

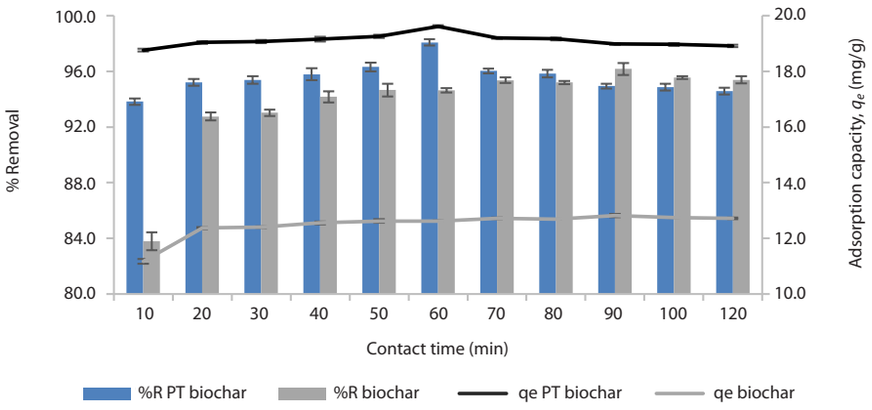

Figure 6 Effect of contact time [Conditions: pH = 2 (Untreated biochar), 12 (PT biochar); Dosage : 3 g/L (Untreated biochar), 2 g/L (PT biochar); Concentration = 40 mg/L (both biochars); Temperature = 25°C]

achieved a maximum q_e of 12.8 mg/g at 90 min, while the PT biochar reached a higher q_e of 19.6 mg/g within 60 min. The faster equilibrium of the PT biochar indicates improved adsorption kinetics, likely due to enhanced surface functional groups and increased pore accessibility. After equilibrium, the adsorption capacity values remained nearly constant, reflecting surface saturation and stable adsorption for both biochars.

CONCLUSION

This study investigated the potential of banana pseudo-stem biochar, especially when modified through acid pre-treatment, as an effective adsorbent for removing crystal violet dye from aqueous solutions. Batch adsorption experiments showed that both untreated and acid pre-treated biochar efficiently removed crystal violet across various operating parameters, with PT biochar consistently exhibiting superior adsorption performance. The optimal conditions for maximum dye removal were found to be pH 2 for untreated biochar and pH 12 for PT biochar, with optimal doses of 3 g/L and 2 g/L, respectively. The highest removal efficiency occurred at an initial CV concentration of 40 mg/L and contact times of 90 min for untreated biochar and 60 min for PT biochar. At the optimum adsorption conditions, the adsorption capacities of PT biochar and untreated biochar were 19.6 mg/g and 12.8 mg/g, respectively. Acid modification notably improved the adsorption capacity of PT biochar by increasing surface area, surface functional groups, and pore structure. These enhancements strengthen dye-adsorbent interactions and accelerate adsorption kinetics. Future research could focus on adsorption isotherms, kinetics, and regeneration studies of the modified acid-treated banana pseudo-stem, as well as its practical application in real industrial effluents.

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