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ORIGINAL ARTICLE

Regeneration of *Pinus patula*-derived biochar employed in crystal violet removal in water.

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Abstract

Biochar (BC) is a promising adsorbing material used to eliminate different contaminants from water, including dyes. However, the applicability and cost-effectiveness of this adsorbent also depend on its regeneration capacity. Several approaches to BC regeneration are known, each yielding variable efficiencies, risks of generating secondary pollution, difficulties of application, and contrasting cost/benefit ratios. Chemical regeneration is one of the adsorbent regeneration techniques yielding the best results in contaminant desorption from the BC surface. The present study evaluated the regeneration of *Pinus patula* pellet-derived BC employed in Crystal Violet (CV) adsorption from water using several acids, bases, and solvents. The tested desorption agents included solutions of hydrochloric acid (HCl) 0.1 M, acetic acid (CH_3COOH) 1 M, 75 % ethanol (C_2H_6O), and two 95:5 mixtures of ethanol (75 % and 96 %) and concentrated acetic acid. The 95:5 mixture of 96 % ethanol-concentrated acetic acid was the best-performing solvent with a desorption efficiency of 21.1 %. Therefore, it was chosen as the desorption agent and used for six consecutive adsorption-desorption cycles. After these cycles, the BC removal efficiency decreased up to 54.4 %. This work revealed that the BC used has a good regeneration capacity, which could reduce the costs of implementing water decontamination process having a positive impact on sustainable development by aligning with circular bioeconomy principles.

Keywords: biochar; desorption; reutilization; wastewater.

1. Introduction

Adsorption is one of the most employed methods in water dye removal due to its high efficiency, availability of different adsorbent materials, and simple operation [1]. Adsorption advantages rely on aspects such as adsorbent regeneration capacity. This feature determines technique applicability and drives generation cost reductions, aiming at more efficient and competitive materials [2]. Regeneration is conducted to recover the initial adsorption capacity of a material by eliminating the contaminant molecules retained on its surface. Therefore, the regeneration of a spent adsorbent can improve the application cost of the process and decrease the formation of associated sludge [3].

Adsorbent regeneration studies involve the desorption or decomposition of contaminant molecules retained on the adsorbent surface [2]. To evaluate the regeneration capacity of an adsorbent, various adsorption-desorption cycles must be completed, determining the number of cycles occurring without substantially losing the material's removal efficiency [4]. The technologies that have been used to regenerate adsorbents include the use of reagents, supercritical fluids, microwave irradiation, and thermal regeneration [5].

Adsorbents including activated carbon, clay, nanoparticles, sewage sludge, and biochar have been used for removing dyes in water. Among these, biochar stands out due to its physicochemical characteristics such as porosity, functional groups, and specific surface areas [6–8]. Additionally, biochar has been described as one of the most economically feasible and effective adsorbing materials in water dye removal [6–8]. Biochar is produced from the thermal decomposition of waste biomass like agricultural and industrial by-products, wood, invasive plants, food waste, sewage sludge, animal manure, etc. [9]. Biochar production is an approach to wealth strategy, providing a route for waste management and environmental degradation [10]. For example, wood pellets are biomass forms used in biochar production, which can be subsequently employed in water decontamination [11–13]. In addition to biochar's contribution to wealth and its efficiency in removing dyes, biochar has demonstrated a good regeneration capacity, being able to be used in different adsorption-desorption cycles [14–16]. Therefore, biochar has been described as a promising material to help achieve sustainable development goals and circular economy, especially in wastewater treatment [17, 18].

Biochar, as adsorbent material, is commonly regenerated through chemical processes, focusing on breaking the adsorption equilibrium existing between the adsorbent (biochar) and the adsorbate (dye). For this purpose, different reagents have been used, such as sodium hydroxide (NaOH), acetic acid (CH_3COOH), hydrochloric acid (HCl), methanol (CH_3OH), and ethanol (C_2H_6O) [19].

Under this scenario, the present work addressed the regeneration of biochar from the gasification of *Pinus patula* pellets employed to eliminate Crystal Violet (CV) from water. Chemical regeneration assays were conducted, evaluating the desorption capacity of organic and inorganic solvents to obtain the solution with the highest desorption capacity. Additionally, we assessed the number of biochar desorption-adsorption cycles achieved without losing its removal capacity.

2. Materials and Methods

2.1. Reactants

CV (purity of 99 %), supplied by Carlo Erba (Spain), was used as the pollutant of interest. Additionally, sodium hydroxide (purity > 99 %) and hydrochloric acid (37.30 % pure) were obtained from Sigma Aldrich (USA) and VWR Chemicals (USA), respectively. Acetic acid (99.85 % pure) and ethanol (96 % pure) were procured from a local supplier. Distilled water was used for preparing the reactants solutions. All these reactants and chemicals were used without further purification.

2.2. Biochar production and characteristics

Pinus patula wood pellets were transformed into biochar through gasification at atmospheric pressure. The pellets were obtained from a local sawmill in Medellín (Colombia) with lengths and diameters of 10–15 and 8 mm, respectively. For the gasification procedure, a top-lit updraft reactor was employed [20]. The reactor had a cylindrical shape with a height of 0.28 m and an inner diameter of 0.16 m. The temperature within the gasification bed was monitored by thermocouples (5K-type) (±1 °C), which were each 0.04 m in length throughout the reactor. The gasification agent was atmospheric air, which was supplied to the reactor from a reciprocating air compressor with a flow up to 254 L/min and operated at 300 rpm and 2.60 kW. Airflow and pressure were controlled with a rotameter and a manometer. The air injected into the reactor flowed at 146 ± 4.35 L/min. Data were acquired and managed with the LabView® software and

a National Instruments acquisition card (DAQ) (USB-6001). The wood pellets were heated in the top part of the reactor at approximately 700 °C and dropped from the reaction front to the reactor cover on the bottom [20]. **Table 1** shows the employed gasification conditions.

Table 1. Gasification parameters used in *Pinus patula*-derived biochar production.

Parameter	**Value**
Biomass-air equivalence ratio (Fr)	1.52 ± 0.19
Specific consumption rate of the biomass (m_{bms}) ($kg/h/m^2$)	125.33 ± 15.48
Biochar yields (Y_{char}) (wt%)	12.12 ± 1.19

As presented by Gutiérrez *et al*. [20] and revealed in **Table 2**, after *Pinus patula* wood pellet gasification, the volatile material (VM) decreased, creating biochar surface pores, enhancing its porous structure, and increasing its surface area compared to that of the raw wood [12, 20, 21]. During biochar generation at high temperatures, biomass organic components carbonize, resulting in biochar with more meso- and micropores and a higher surface area, which enhances this material's adsorption [22].

Biochar oxygen and hydrogen to carbon (O/C and H/C) rates are inversely proportional to hydrophobicity and aromaticity [23]. Raw biomass O/C and H/C values decreased after biomass transformation into biochar, indicating that the biochar has a hydrophobic and aromatic surface [24], as expected since the biochar used was derived from lignin-rich biomass [5]. The concentration reduction of O and H follows the loss or modification of functional groups that are thermally changeable, including aliphatic (-CH_2-), -OH, cellulose-like O-alkyl (C-O), and carboxyl (-COOH) groups, and the conjugated graphitic domain formation resulting from aromatic structure condensation [23, 25, 26]. The hydrophobic surface of biochar is also related to the semi-volatile organic compounds on its surface [27].

Table 2. Physicochemical characteristics of *Pinus patula* pellets and biochar.

Physicochemical parameter	**Wood pellets**	**Biochar**
Volatile Material (VM, wt%)	84.64	20.59
Ash Content (AC, wt%)	1.27	1.92
Fixed Carbon Content (FC, wt%)	14.09	77.49
Oxygen (O, wt%)	47.28	0.90
Nitrogen (N, wt%)	0.02	0.19
Hydrogen (H, wt%)	5.69	0.97
Carbon (C, wt%)	47.01	97.94
O/C	0.75	0.01
H/C	1.45	0.12
Surface area (m^2/g)	1.16	367.33
ϱ (kg/m^3)	559.97	236.28
pH	-	8.80
CEC (meq/100 g)	-	21.70

2.3. Desorption experiments

Before studying the desorption efficiency of the chosen reagents reported in the literature, adsorption studies were conducted using a CV solution of 25 mg/L and optimal operating conditions. The concentration of CV in textile wastewater can vary from 1 mg/L to 1000 mg/L. Additionally, the CV level in water employed in removal studies ranges between 20 mg/L and 600 mg/L [28–32]. The CV experimental adsorption process conditions were 13.5 g/L of biochar (300–450 μm of particle size) and a solution pH of 9 in 200 mL of water containing CV. A retention time of 3 min was applied because it is the time span adsorption equilibrium is reached. After 3 min, 10 mL aliquots were filtered using 0.45 μm nylon filters. A spectrophotometer DR 2700 model (Hach, USA) was used for determining the CV concentration in water at a λ of 582 nm. The remaining CV level in the solution after biochar-based treatment was determined with a calibration curve ($R^2 = 0.9973$).

Previously conducted adsorption studies revealed that electrostatic attraction was one of the adsorption mechanisms involved [33]. According to several studies addressing *Pinus patula* biochar-driven CV removal optimization, basic media (pH = 9) favors CV adsorption. Thus, to trigger desorption, biochar spent with CV was immersed in a 0.1 M hydrochloric acid solution for 30 min and stirred at 200 rpm [34]. Alternatively, the spent biochar was immersed and stirred at 200 rpm for 30 min in 100 mL of 75 % ethanol [35], and Park and coworkers employed a mixture of methanol and acetic acid [36]. However, methanol is potentially toxic since it can lead to formaldehyde (CH_2O) production when digested [37]. To overcome this possible disadvantage, in the present work's desorption assays, ethanol replaced methanol because of its reported similar regeneration performances and reduced toxicity potential [35]. Hence, the modified and employed method consisted of immersing the spent biochar in a 95:5 75 % ethanol and concentrated acetic acid mixture (C_2H_6O:CH_3COOH) for 30 min at a stirring speed of 200 rpm. This ethanol-acetic acid mixture was also assessed by increasing the ethanol concentration from 75 % to 96 %.

After the desorption assays, aliquots were taken and analyzed by visible light spectrophotometry to calculate the concentration of CV desorbed from the biochar surface. The desorption efficiency was calculated according to Eq. (1) [34], where D (%) and q_{des} (mg/g) are the solvent desorption efficiency and capacity, respectively, and q_{ads} (mg/g) is the biochar's adsorption potential. In turn, to calculate q_{ads} and q_{des}, Eqs. (2) and (3) were used. In these Eqs., C_i and C_f (mg/L) are CV's initial and final concentrations; m (g) is the biochar mass; V (L) is the volume of the solution used in the adsorption process; C_s (mg/L) is the level of CV in the solvent; and V_s (L) is the solvent volume used in the desorption process [3].

$$D(\%) = \frac{q_{\text{des}}}{q_{\text{ads}}} \times 100 \tag{1}$$

$$q_{\text{ads}} = \frac{(C_i - C_f)V}{m} \tag{2}$$

$$q_{\text{des}} = \frac{C_s V_s}{m} \tag{3}$$

2.4. Regeneration experiments

Once the desorption efficiencies of 0.1 M hydrochloric acid, 75 % ethanol, and the 95:5 ethanol: acetic acid solution were evaluated, the solvent with the highest desorption efficiency was selected for the regeneration studies. The number of desorption-adsorption cycles a biochar sample can endure without losing its maximum removal efficiency was studied following the adsorption and desorption assay conditions above. The *Pinus patula* biochar was separated from the treated solution using a paper filter with < 375 μm of pore diameter. Subsequently, the CV-depleted biochar was dried at room temperature for 12 h. After biochar regeneration with the selected solvent, the latter was removed with a paper filter (pore diameter < 375 μm), and the biochar dried at room temperature for 12 h. Tests were run in triplicate, and the regeneration treatment was performed until biochar removal efficiency fell by approximately 50 %.

3. Results and discussion

3.1. CV removal efficiencies

The first adsorption cycle in a 25 mg/L CV solution operated at optimal conditions (pH 9, biochar dose of 13.5 g/L, and 300–450 μm of biochar particle size). The observed CV removal efficiency was > 99.9 %, as shown in **Fig. 1**. The high elimination efficiency of CV by biochar derived from *Pinus patula* pellets revealed that the adsorbent is effective in removing CV in water [38]. Fig. 1 reveals a removal process characterized by a high adsorption rate reaching a removal efficiency of 99.8 % shortly after 1 min of retention time. At 3 min of treatment, the adsorption equilibrium was reached, and the CV removal was maintained at approximately 99.9 %. The fast adsorption rate is associated with the existence of active sites for adsorption on the biochar surface and the affinity between CV molecules and the active sites for adsorption in the *Pinus patula* biochar [34]. Consequently, the CV removal from water takes place during the first minutes of contact with *Pinus patula* biochar.

When the adsorption process of a dye is promoted in an acid or basic medium, an alteration in the solution pH disrupts the adsorption equilibrium between the adsorbent surface and the dye molecule due to an increase in the concentration of OH^- or H^+, triggering dye desorption [39]. CV adsorption by biochar is favoured at pH 9; therefore, a 0.1 M hydrochloric acid, solution was evaluated as the desorbing agent. The effect of this solution, as observed in **Fig. 2**, was negligible given its desorption efficiency of 0 %. The desorption efficiency of 1 M acetic acid was also evaluated, reaching a desorption efficiency of 0.12 %. These results are a likely consequence of multiple mechanisms governing the stable adsorption of CV molecules on biochar surfaces other than electrostatic attractions [33]. Therefore, as reveled by these results, the sole manipulation of such electrostatic attractions does not trigger biochar regeneration.

In turn, the desorption efficiency obtained with a 75 % ethanol solution was 3.3 % (Fig. 2). Considering that the adsorption principle is based on adsorbate and adsorbent affinity, the observed desorption of 75 % ethanol can be associated with the high affinity between CV molecules and *Pinus patula* biochar. Therefore, this solvent is insufficient to disrupt the adsorption equilibrium between the biochar and the dye molecules [40]. Additionally, CV removal by biochar was mainly due to chemical adsorption mechanisms. In this regard, the employed ethanol solution is unable to produce enough energy to break the bonds between the biochar active sites and CV molecules [3].

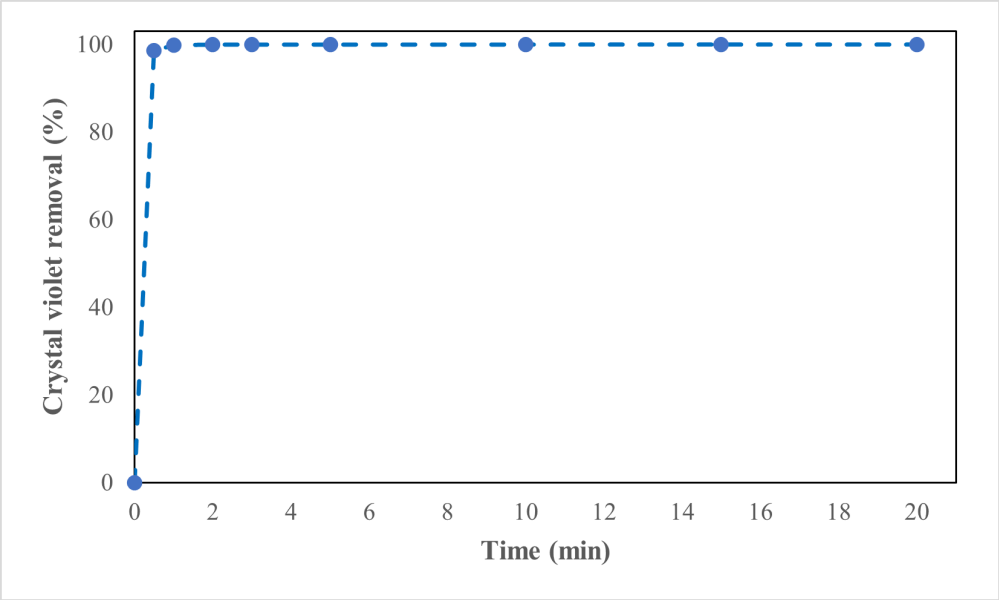

Figure 1. Removal efficiency of Crystal Violet (CV) by *Pinus patula*-derived biochar. Operating conditions: biochar concentration = 13.5 g/L, CV concentration = 25 mg/L, biochar particle size = 300-450 μm, pH = 9 and residence time = 3 min.

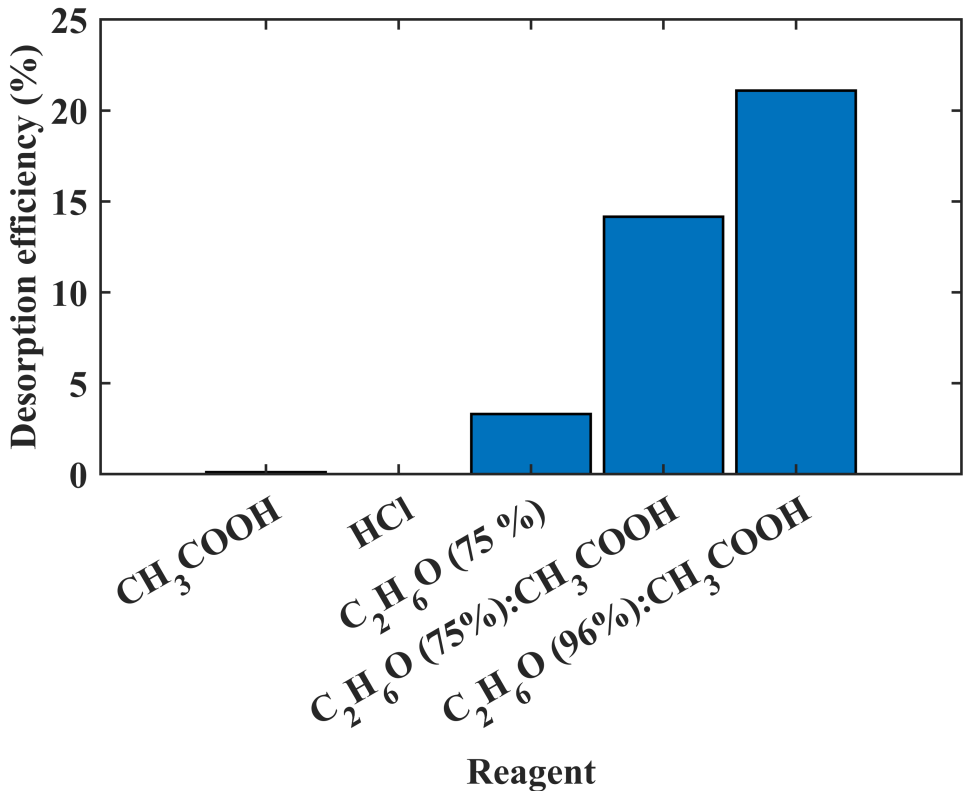

Figure 2. Desorption efficiencies of five reagents (acetic acid, CH_3COOH; hydrochloric acid, HCl; 75 % ethanol, C_2H_6O (75 %); 75 % ethanol-acetic acid, C_2H_6O (75 %) : CH_3COOH; 96 % ethanol-acetic acid, C_2H_6O (96 %) : CH_3COOH) on CV-spent *Pinus patula* biochar.

The assesed 95:5 ethanol (75 % / 95 %) -acetic acid solutions yielded desorption efficiencies of 14 % and 21 %, respectively (Fig. 2). The improved desorption efficiency of the tested ethanol-acetic acid solutions can be attributed to thier capacity to influence the chemical bonds and electrostatic attractions underlying the adsorption mechanism [16]. These reagents in combination broke some of the bonds created between the CV and the biochar, leading to higher desorption efficiencies than those of media contiaing either acids or ethanol alone. Moreover, the enhanced desorption efficiency observed when rising the ethanol concentration from 75 % to 96 % in the ethanol-acetic adic solution is attributed to the fact that solvent higher concentrations usually result in better dye molecules desorptions [16].

Based on the desorption results obtained, the 95:5 ethanol (96 %)-acetic acid solution was selected for the regeneration studies. However, the overall low desorption efficiencies achieved in this study revealed the presence of strong adsorption forces between CV and the *Pinus patula* biochar, being mainly influenced by internal internal pore diffusion and $\pi - \pi$ electron interactions. This means that the pathway the dye molecules need to follow for desorption from the biochar surface is difficult to reach and that π-π electron interactions, which are strong bonds, are difficult to break [36]. These results agree with those reported for methylene blue [16].

3.2. Regeneration studies

Once exhausted with CV, the biochar generated during *Pinus patula* pellet gasification was regenerated using a 95:5 ethanol (96 %)-acetic acid solution. Experimental adsorption-desorption cycles proceeded with an initial CV level of 25 mg/L, a pH of 9, and a retention time of 3 min. Biochar removal efficiency decreased from 98.8 % to 54.4 % after six complete adsorption-desorption cycles, as depicted in **Fig. 3**. The decrease in biochar removal efficiency may be ascribed to adsorption active site saturation by CV molecules, gradually depleting available active sites [41]. As the adsorption-desorption cycles progressed, biochar weight decreased, reflecting active site number reductions. This phenomenon was also observed by Mensah and coworkers [42]. Furthermore, because desorption efficiency was relatively low, some CV molecules were not desorbed from the biochar surface, causing active site blockage [43].

The observed *Pinus patula* biochar performance during its regeneration assays revealed its stability as an adsorbent and its capability to be regenerated and used in multiple desorption-adsorption cycles. In this regard, the production and application costs to treat dye-polluted water align with sustainable development and circular bioeconomy [44]. The cost of biochar production can vary between 100 and 600 USD/Ton depending on the thermochemical process used in biochar generation. Producing biochar is less expensive than activated carbon, considering that one Ton of commercial activated carbon can cost 800 to 1000 USD [45, 46]. In addition to having a lower production cost, the regeneration capacity of biochar makes it an adsorbing material that stands out in comparison with activated carbon, resulting in operational cost reductions [47]. The reagents used in the present study are widely available. Moreover, considering the volume used for each reagent, the cost associated with the biochar regeneration was 4.71 USD, demonstrating the cost-effectiveness and scalability of biochar regeneration when applied to remove dyes from water.

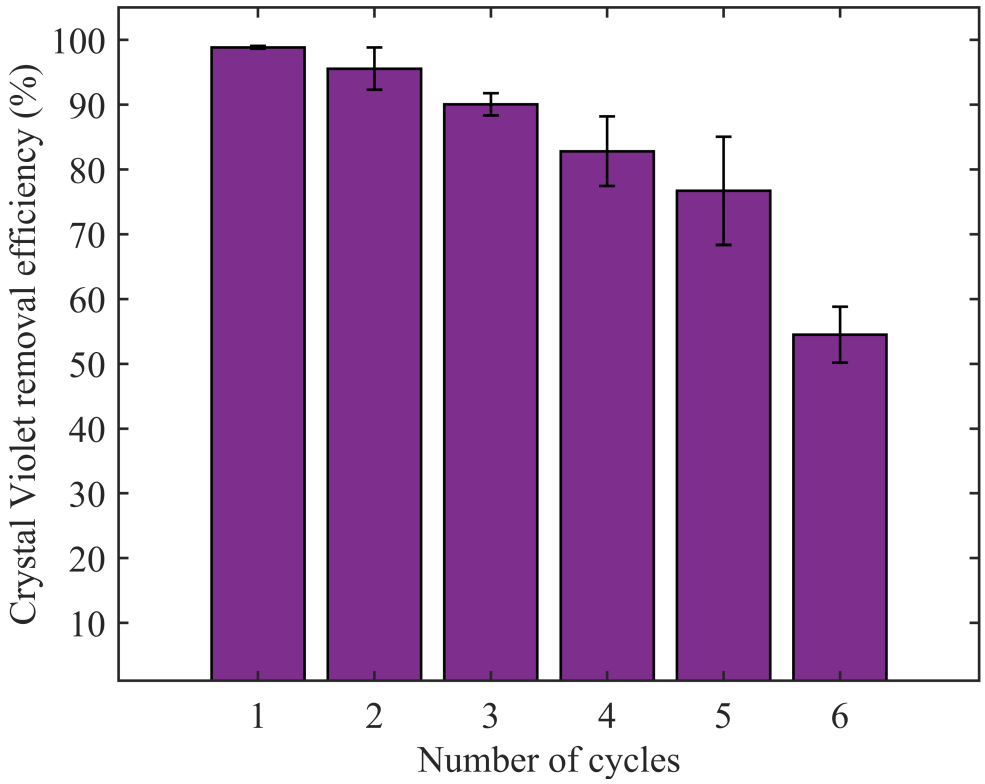

Figure 3. Crystal Violet (CV) adsorption-desorption cycles by the biochar. Operating conditions: biochar particle size = 300-450 μm, biochar concentration = 13.5 g/L, CV concentration = 25 mg/L, pH = 9 and residence time = 3 min.

4. Conclusions

The low desorption efficiency observed in this study with different types of solvents revealed that CV adsorption into *Pinus patula* biochar was strong and dominated by electron π interactions and internal pore diffusion. Considering this caveat, the best-performing solution for CV desorption was the 95:5 96 % ethanol-acetic acid solution. Additionally, we determined that the adsorbent (13.5 g/L) may be utilized for up to six desorption-adsorption cycles with an initial CV concentration of 25 mg/L before the removal efficiency drops to approximately 50 % of that observed in the first cycle. *Pinus patula* biochar has good recyclability since it can endure different adsorption-desorption cycles.

Further studies should be conducted evaluating other biochar saturation and regeneration methods to develop processes with higher desorption efficiencies, improved cost-benefit, and minimal probabilities of secondary pollution. Additionally, further studies on the performance of *Pinus patula* biochar in the continuous removal of CV should help to determine adsorbent stability.

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6. Conflict of interest

The authors declare no conflict of interest.

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Regeneración de biocarbón derivado de *Pinus patula* empleado en la remoción de violeta de cristal en agua.

Resumen: El biocarbón (BC) es un material adsorbente prometedor utilizado para eliminar diversos contaminantes, incluyendo colorantes, del agua. No obstante, la aplicabilidad y rentabilidad de este adsorbente dependen de su capacidad de regeneración. Se conocen diversas aproximaciones para regenerar el BC, cada una asociada a diferencias en eficiencia, riesgos de generar contaminación secundaria, dificultades de aplicación y relaciones costo-beneficio. La regeneración química es una de las técnicas más eficaces para desorber contaminantes de la superficie del BC. El presente estudio evaluó la regeneración de BC derivado de pélets de *Pinus patula*, empleado en la adsorción de violeta de cristal (CV) del agua, utilizando varios ácidos, bases y disolventes. Los agentes de desorción probados incluyeron soluciones de ácido clorhídrico (HCl) 0,1 M, ácido acético (CH_3COOH) 1 M, etanol (C_2H_6O) al 75 %, y dos mezclas (95:5) de etanol (75 % y 96 %) con ácido acético concentrado. La mezcla 95:5 de etanol al 96 % con ácido acético concentrado fue el disolvente más eficiente, alcanzando una eficacia de desorción del 21,1 %. Por ello, se seleccionó como agente de desorción y se empleó durante seis ciclos consecutivos de adsorción-desorción. Tras estos ciclos, la eficiencia de remoción del BC se redujo a un nivel de hasta el 54,4 %. Este estudio evidenció que el BC utilizado posee una buena capacidad de regeneración, lo que podría reducir los costos de implementación en procesos de descontaminación de agua, contribuyendo al desarrollo sostenible y alineándose con los principios de la bioeconomía circular.

Palabras Clave: Aguas residuales; Biocarbón; Desorción; Reutilización.

Regeneração de biocarvão derivado de Pinus patula utilizado na remoção de violeta de cristal em água.

Resumo: O biocarvão (BC) é um material adsorvente promissor utilizado para remover diversos contaminantes, incluindo corantes, da água. No entanto, a aplicabilidade e a viabilidade econômica desse adsorvente dependem de sua capacidade de regeneração. Existem diversas abordagens para regenerar o BC, cada uma associada a diferenças em eficiência, riscos de gerar poluição secundária, dificuldades de aplicação e relações custo-benefício. A regeneração química é uma das técnicas mais eficazes para promover a dessorção de contaminantes da superfície do BC. O presente estudo avaliou a regeneração de BC derivado de pellets de *Pinus patula*, utilizado na adsorção de violeta de cristal (CV) da água, empregando diferentes ácidos, bases e solventes. Os agentes de dessorção testados incluíram soluções de ácido clorídrico (HCl) 0,1 M, ácido acético (CH_3COOH) 1 M, etanol (C_2H_6O) a 75 %, e duas misturas (95:5) de etanol (75 % e 96 %) com ácido acético concentrado. A mistura 95:5 de etanol a 96 % com ácido acético concentrado foi o solvente mais eficiente, alcançando uma eficiência de dessorção de 21,1 %. Por esse motivo, foi escolhida como agente de dessorção e utilizada em seis ciclos consecutivos de adsorção-dessorção. Após esses ciclos, a eficiência de remoção do BC reduziu para até 54,4 %. Este estudo demonstrou que o BC utilizado possui uma boa capacidade de regeneração, o que pode reduzir os custos de implementação em processos de descontaminação da água, contribuindo para o desenvolvimento sustentável e alinhando-se com os princípios da bioeconomia circular.

Palavras-chave: Águas residuais; Biocarvão; Dessorção; Reutilização.

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