Preparation of low-cost adsorbents from Bidens pilosa L. weed using H3PO4 and KOH as activating agents and assessment of their organic dyes adsorption

Preparación de adsorbentes de bajo costo a partir de la maleza Bidens pilosa L. utilizando H3PO4 y KOH como agentes activadores y evaluación de su capacidad de adsorción de colorantes orgánicos

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ABSTRACT

The present study produced activated carbons (AC) derived from chemically treated Bidens pilosa weed (BPW) through traditional pyrolysis. The ACs were used to treat dyes, such as rhodamine B (RhB), methyl orange (MO), and methyl red (MR), from aqueous solutions. The influence of impregnated ratios (1:0.5, 1:1, 1:1.5, and 1:2) of potassium hydroxide (KOH) and phosphoric acid (H3PO4) as chemical activation agents on adsorption efficiency was investigated. The results showed that for all investigated dyes, the carbon activated by the H3PO4, agent at an impregnated ratio of 1:2 (ACH4) exhibited the best adsorbent for dye removal. After 300 min of uptake experiments, the adsorption reached equilibrium, with adsorption efficiencies of 98.6, 95.5, and 96.0% for RhB, MO, and MR, respectively. ACH4 was analyzed using Fourier-transform infrared spectroscopy (FTIR) spectra and scanning electron microscopy (SEM). The SEM image revealed a progressive development of pores resulting from removing volatile substances and impurities. FTIR analysis indicated the predominance of acidic surface functional groups, which favored the adsorption process. Therefore, ACH4 prepared from Bidens pilosa weed is a promising adsorbent for removing dyes from an aqueous solution.

Keywords: *Bidens pilosa*, activated carbon, dye removal, adsorption, chemical activation.

RESUMEN

En el presente estudio, se produjeron carbones activados (CA) derivados de la maleza Bidens pilosa (BPW) tratada químicamente mediante pirólisis tradicional. Los CA se utilizaron para tratar colorantes, por ejemplo, rodamina B (RhB), naranja de metilo (MO) y rojo de metilo (MR) a partir de soluciones acuosas. La influencia de las proporciones de impregnación (1:0.5, 1:1, 1:1.5, y 1:2) de hidróxido de potasio (KOH) y ácido fosfórico (H_3PO_4) como agentes químicos de activación sobre la eficiencia

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de adsorción fue investigada. Según los resultados, para todos los colorantes investigados, el carbón activado por el agente H_3PO_4 en una proporción de impregnación de 1:2 (ACH_4) presentó el mejor adsorbente para la eliminación del colorante. Transcurridos 300 min de experimentos de adsorción, la adsorción alcanzó el equilibrio, con eficiencias de adsorción del 98,6, 95,5 y 96,0% para RhB, MO y MR, respectivamente. Se analizó el ACH_4 mediante espectroscopia infrarroja con transformada de Fourier (FTIR) y microscopia electrónica de barrido (SEM). La imagen SEM reveló un desarrollo progresivo de poros resultante de la eliminación de sustancias volátiles e impurezas. El análisis FTIR indicó el predominio de grupos funcionales ácidos en la superficie, lo que favoreció el proceso de adsorción. Por lo tanto, el ACH_4 preparado a partir de la maleza Bidens pilosa es un adsorbente prometedor para la eliminación de colorantes de soluciones acuosas.

***Palabras clave**: Bidens pilosa, carbón activado, eliminación de colorantes, adsorción, activación química.*

INTRODUCTION

Water pollution is a pressing global concern, exacerbated by various pollutants, including organic dyes that resist degradation, especially in the textile industry. Rhodamine B (RhB), a common cationic dye, is water-soluble and toxic to humans and animals upon contact. Methyl orange (MO), an anionic dye, exhibits mutagenic properties. In contrast, methyl red (MR), a non-ionic dye, is mutagenic, carcinogenic, and persistent in aquatic environments, emphasizing the need for effective treatment and elimination of these harmful dyes to mitigate their impact [1]-[4]. Due to its porous structure and abundant functional groups, activated carbon (AC) has emerged as a highly effective adsorbent for wastewater treatment. However, the limited affinity for larger contaminants associated with commercial activated carbons (CAC) has led researchers to explore the production of AC from low-cost precursors, such as weed biomass, which urgently need of environmental protection and ecological sustainability [5]-[8].

Bidens pilosa L. (BP) is a globally invasive weed that poses a substantial ecological threat through the displacement of native vegetation, disruption of ecosystems, and economic losses, ultimately contributing to a decline in biodiversity losses [9], [10]. Hence, this investigation is situated within the context of valorizing this harmful grass by producing porous materials, specifically activated carbon, utilizing chemical activation methods involving phosphoric acid (H_3PO_4) and potassium hydroxide (KOH). These chemical activators are widely used to activate the biochars because they can react with the carbon of biochars to develop new pores, supply more functional groups, and increase surface area. Subsequently, the efficacy of these materials in removing RhB, MO, and MR from water was assessed. The porous carbons' physicochemical attributes were assessed through scanning electron microscopy (SEM) and Fourier-transform infrared reflection (FTIR) analyses.

MATERIALS AND METHODS

Materials

The chemicals (purity > 98%) H_3PO_4 and KOH were purchased from Scharlau, Spain, and were employed as the activating agents during the material synthesis activation process. RhB, MO, and MR were procured from Sigma Aldrich, Singapore, and were selected as the model molecules to assess the carbon adsorption efficiency of the carbons. All substances utilized in the experimental procedures met the standards of analytical-grade reagents.

AC preparation

The dried material of the invasive weed *Bidens pilosa* L. (BP) was crushed and powdered, then carefully loaded on ceramic crucibles and heated in a muffle furnace under an air-deficient ambient at 400 ºC for 180 min to form biochar (BC).

The BP was chemically activated with an activating agent, H_3PO_4 or KOH. In detail, the BP samples were soaked in the desired activating agent with impregnation ratios of 1:0.5, 1:1, 1:1.5, and 1:2 (chemical agent (g): biomass (g)). Next, 5 mL of deionized water was added to each impregnated BP sample. The mixtures were mixed well and left at temperature for six hours before transferring them to the oven. The samples were dried at 100 ºC for six hours to remove any remaining moisture.

The impregnated samples were carbonized in a horizontal tubular furnace at 600 ºC for three hours under nitrogen gas (N_2) with a heating rate of 5 ºC/min. The obtained activated carbons (AC) were washed to neutrality, dehydrated at 80 ºC for six hours, ground, and stored. They were labeled ACH-(1,2,3,4) and ACK-(1,2,3,4) for the impregnation ratios of 1:0.5, 1:1,1:1.5, and 1:2 with H_3PO_4 and KOH, respectively.

Characterization

The surface chemistry of adsorbents was conducted using Fourier transform infrared (FTIR) spectrometry on the Nicolet 6700 spectrophotometer (Thermo Fischer Scientific Inc., Waltham, MA, USA) in 400-4000 cm^{-1}. The surface morphology of adsorbents was performed by scanning electron microscopy (SEM) on JSM-6510 (JEOL/EO, Tokyo, Japan).

Adsorption test

Batch experiments were conducted to study the dye adsorption efficiency onto synthesized activated carbon samples. A quantity of 6 mg of AC was mixed with 60 mL of dye solution (10 mg/L) at a temperature of 30 ºC; operations conditions were chosen based on the literature [8]. To examine the adsorption of the materials, 2 mL of each sample aliquot will be taken every 60 min (0, 60, 120, 180, 240, 300, 360). Afterward, the adsorbent was separated by centrifugation. The aliquot's concentration in each dye was identified with UV-Vis spectroscopy. Removal or R(%) and capacity or Q(mg/g) were calculated by equation (1) and equation(2).

$$R(\%) = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

$$Q = \frac{C_o - C_e}{C_o} \times V \quad (2)$$

where, C_o and C_e are initial, and equilibrium dye concentrations (mg/L); m (g) and V (L) are AC mass, and dye volume.

RESULT AND DISCUSSION

Vibrational spectroscopy

Figure 1 depicts the Fourier-transform infrared (FTIR) spectra of BC and activated carbon derived from H_3PO_4 activation with an impregnation ratio of 1:2 (ACH4) in the wavelength region from 4000 to 400 cm^{-1}. Notably, discernible differences in spectral features are evident between BC and ACH4, attributed to the activating influence of H_3PO_4. This activation process engenders more functional groups in ACH4 relative to BC. As corroborated by Lian *et al.* [11], principal spectral features of BC encompass peaks at 1700 cm^{-1}, 1250-1180 cm^{-1}, and 785-585 cm^{-1}, associated with vibrational modes of –C–O/C–O–C, aromatic C = C, and C–H moieties, respectively. The peak at 1400 cm^{-1} arises from the vibration of –CH– groups found in aliphatic and aromatic compounds [11], [12]. The presence of peaks at 2310 cm^{-1} signifies the existence of –C≡C– and –C≡N triple bonds in the adsorbents [12]. Additionally, the band centered at 3650 cm^{-1} is ascribed to O–H functional groups (e.g., –OH and –COOH) or hydroxides. Upon subjecting the BC to H3PO4 activation and subsequent pyrolysis, the C-H deformation asymmetric peak intensities, –C≡C– and –C≡N bonds decline in ACH4. Notably, the spectra of ACH4 manifest a peak at 1600 cm^{-1} associated with vibrations within the aromatic ring and a peak at 3440 cm^{-1} corresponding to OH stretching, indicating the presence of analogous functional groups on the surfaces of both BC and ACH4. It is noteworthy that the 1600 cm^{-1} peak assigned to aromatics exhibits greater intensity in ACH4. An additional band within the 960-1200 cm^{-1} region is attributed to P = O stretching, O–C vibrations of the P–O–C (aromatic) bond, or P = OOH functionalities, characteristics that are typically observed in samples subjected to H_3PO_4 treatment [13].

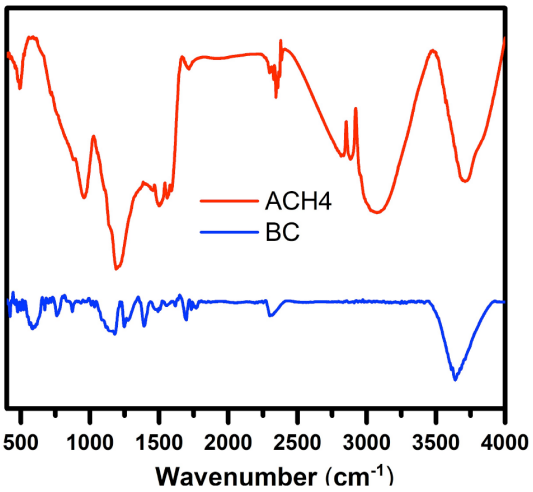

Figure 1. FTIR analysis of BC and ACH4 adsorbents.

Morphological studies

Figure 2 presents scanning electron micrographs (SEM) of both BC and ACH4. Initially, BC exhibits a corrugated surface devoid of pores, as illustrated in Figure 2a. However, following the activation process, subsequent high-temperature carbonization, and thorough washing, the resulting sample manifests a profusion of macropores characterized by diverse sizes and geometries, as depicted in Figure 2b. The employment of H_3PO_4 as an activating agent plays a pivotal role in fostering the development of pores along the length of BC. It is noteworthy that the resultant pores do not assume perfect spherical shapes; instead, they exhibit variations in both length and width within the range of 2 µm to 10 µm, as ascertained through calculations performed using SEM image analysis software H_3PO_4 acts as a cleanser and dehydrating agent, removing tarry residues, promoting bond cleavage reactions, and enabling the formation of protective linkages, which, along with high-temperature carbonization, expedite the development of porosity in the material [14].

The zero-charge point

The zero-charge point (pH_{zcp}) of the ACH4 was determined in this study, which helps to indicate the charge of the adsorbent surface. As can be seen from Figure 3, the pH_{zcp} is found at 2.96. This value means that if the ACH4 adsorbent is immersed in a solution with pH > 2.96, its surface becomes negatively charged and vice versa. The very low pH_{zcp} can be explained by the fact that this ACH4 contains many acidic groups, such as -COOH, $-H_2PO_4$, and others in its structure. The activation with H_3PO_4 may attach these groups on the surface of the ACH4 adsorbent. The same results were reported for the adsorbent derived from *Ziziphus mauritiana* (pH_{zcp} = 3.87) [15] apricot nut shells (pH_{zcp} = 2.13) [16] that were activated by H_3PO_4.

Effect of KOH activation

Figure 4 illustrates the influence of different KOH impregnation ratios on the adsorption efficiency of ACs for RhB, MO, and MR. The adsorption reached equilibrium at 300 minutes for all investigated dyes. Regarding RhB, adding the KOH activation agent significantly reduced the uptake efficiency for all ACKs. Conversely, for MO, all KOH impregnation ratios greatly enhanced the removal capacities of ACs, with ACK1 showing a threefold improvement and ACK3 an eightfold increase compared to BC. In the case of MR, after 300 min of contact time, the uptake efficiency of ACK3 was 74.22%, relatively higher than that of BC (57.61%). Other KOH impregnation ratios resulted in a decrease in MR uptake efficiency. These findings can be elucidated by using impregnated AC with KOH, facilitating the thermal degradation and the volatilization or vaporization of activation temperatures. In turn, this enhances surface area expansion and pore formation, thereby facilitating the ingress of a greater number of dye molecules into the adsorbent.

As compared, Jawad *et al.* [17] successfully transformed dragon fruit peel (DFP) into a highly porous activated carbon (DFPAC) through a KOH-activation process with an impregnation ratio of 1:2. The surface morphology of DFPAC exhibited an irregular, highly porous structure reminiscent of a sponge, which proved to be exceptionally

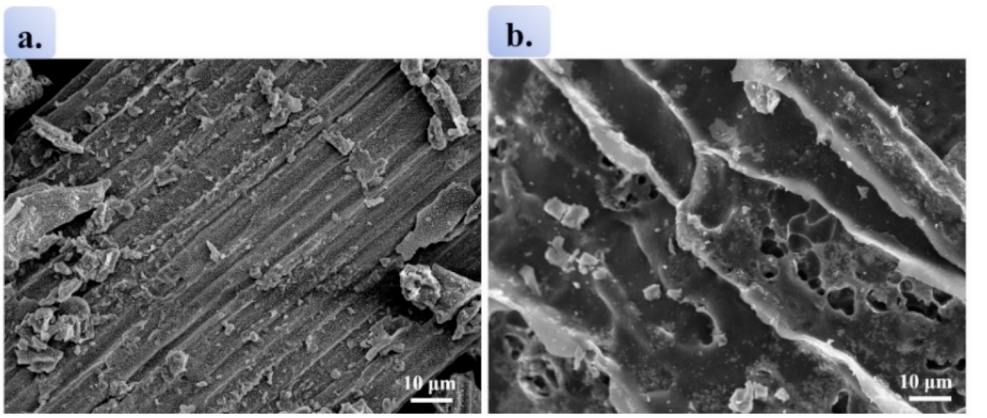

Figure 2. SEM analysis of a) BC and b) ACH4 adsorbents.

well-suited for capturing aromatic dye compounds. Furthermore, the surface area of DFPAC increased by approximately 1575-fold compared to unmodified DFP. These enhancements enabled DFPAC to be an effective adsorbent for removing MB dye, exhibiting a maximum adsorption capacity of 195.2 mg g^{-1}. Nevertheless, the improper introduction of an extra activation agent impaired certain micropores, causing their collapse or fusion, thereby diminishing the surface area and total pore volume [18].

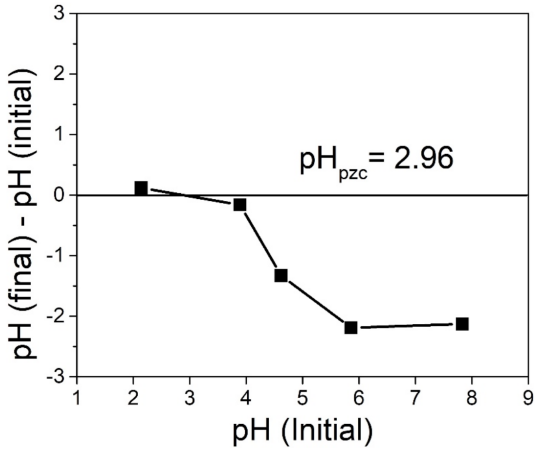

Figure 3. The zero-charge point of the ACH4 adsorbent.

A study conducted by Mariana *et al.* [19] investigated the impact of varying KOH impregnation ratios (1:0.25, 1:0.5, and 1:1) on the functional properties of ACs derived from *Myristica fragrans* shells. The research revealed that microporosity, surface area, and total pore volume increased when the supplementary activating agent was increased from 1:0.25 to 1:0.5 but declined as the impregnation ratio reached 1:1. Samiyammal *et al.* [20] compared the influence of two different KOH impregnation ratios, specifically 1:1 and 1:4, on the characteristics of ACs from cashew nut shells (CNS) subjected to KOH treatment. The research revealed that the BET surface area of

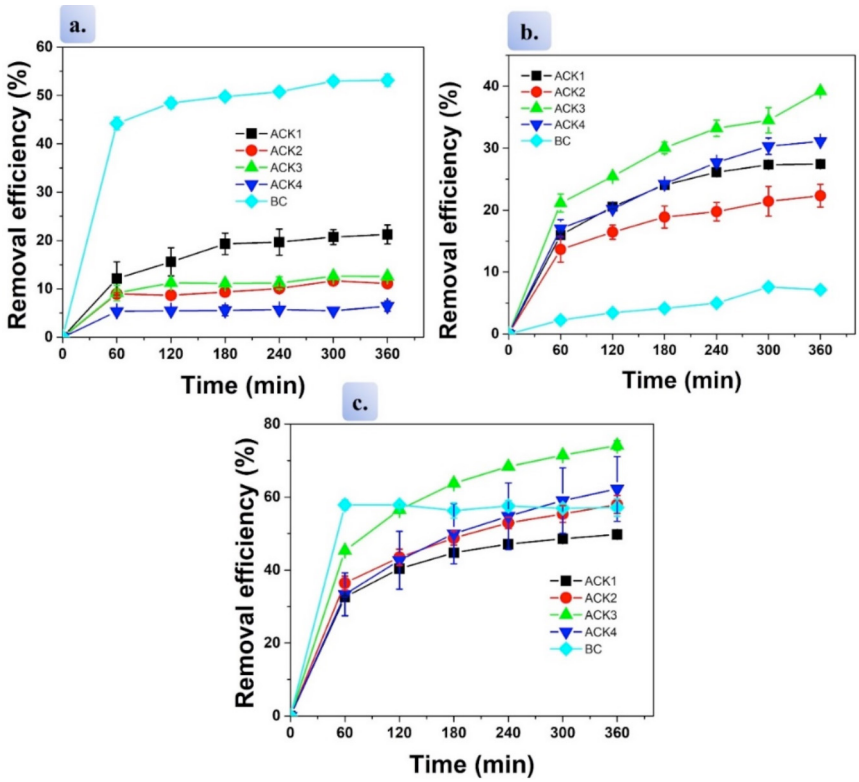

Figure 4. Effect of KOH impregnation ratio on the removal percentages of RhB a) MO b) and MR c) by BP-derived activated carbons. Condition: AC dosage at 0.1 g/L, dye volume at 60 mL, dye concentration at 10 mg/L, temperature at 30 ºC.

CNSAC reached 407.80 m^2 g^{-1} at an impregnation ratio of 1:1 but decreased surface area when the impregnation ratio was altered to 1:4, resulting in a value of 297.10 m^2 g^{-1}. Additionally, SEM images demonstrated the formation of a honeycomb-like porous surface at both impregnation ratios. Notably, the 1:1 impregnation ratio produced a higher number of relatively smaller pores than the 1:4 ratio.

Effect of H3PO4 activation

The impact of the H_3PO_4 impregnation ratio on the removal of RhB, MO, and MR by BP-ACs is depicted in Figure 5. Equilibrium adsorption was also reached after 300 min of contact time for all tested dyes. At this time, the dye removal efficiency of H_3PO_4-activated AC gradually increased as the impregnation ratio went from 1:0.5 to 1:2 in general. This trend was particularly evident for RhB, where the dye uptake efficiency of ACH4 (impregnation ratio of 1:2) was approximately four times higher than that of ACH1 (impregnation ratio of 1:0.5). However, both ACH1 and ACH4 had about half and double the dye adsorption efficiency of BC, respectively (Figure 5a). Conversely, the opposite trend was observed in MO and MR removal, with all ACs exhibiting higher uptake efficiencies than pristine BC. This trend was especially pronounced for MO, as BC had very low adsorption efficiency (7.88%), while ACH1 and ACH2 achieved approximately 54.85% and 89.45%, respectively. However, ACH3 and ACH4 had the same MO removal efficiency (96.36%), slightly higher than ACH2 (Figure 5b).

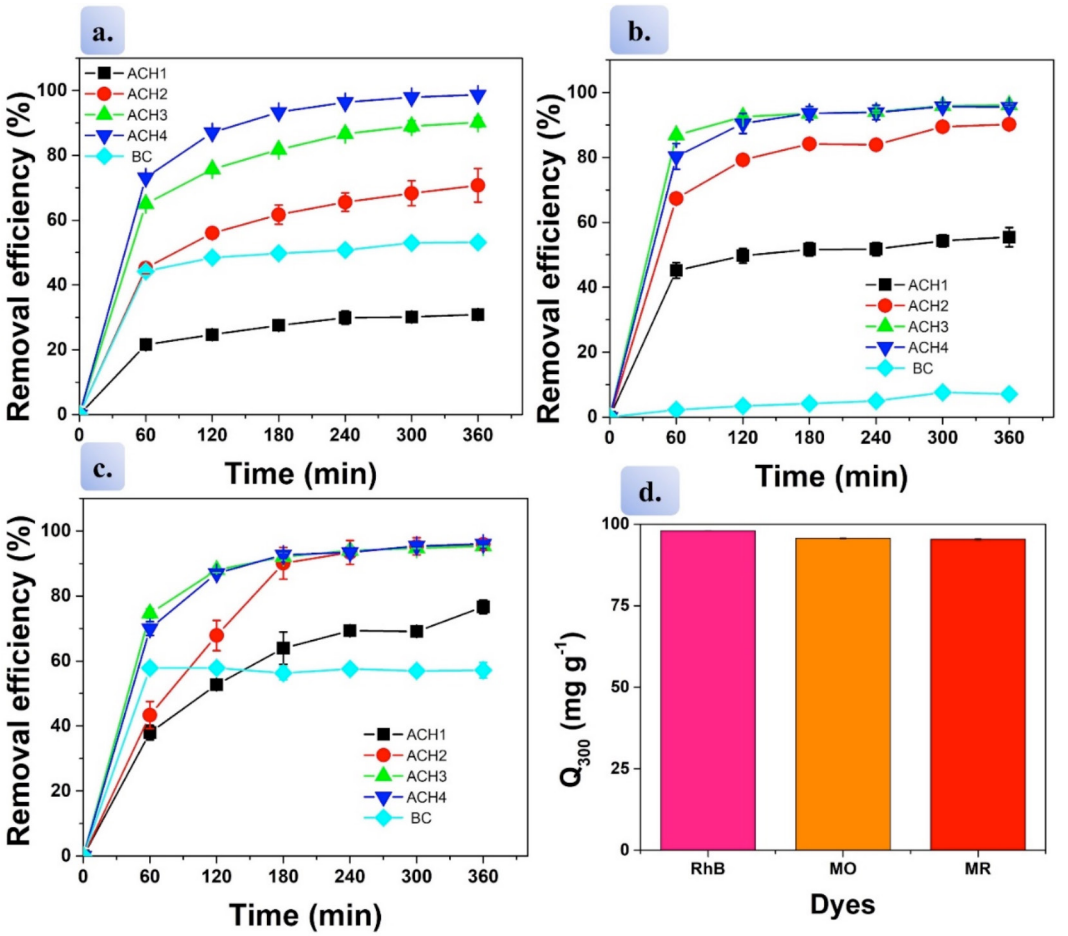

Figure 5. Effect of H_3PO_4 impregnation ratio on the removal percentage of RhB a) MO b) and MR c) by BP-derived activated carbon. The adsorption capacity of three dyes was compared using an ACH4 adsorbent after 300 min d) Condition: AC dosage at 0.1 g/L, dye volume at 60 mL, dye concentration at ten mg/L, temperature at 30 ºC.

In the case of MR, the three ACs mentioned above have nearly identical adsorption efficiencies (~97%), which were almost 16% higher than that of ACH1. These results highlight the crucial role of chemical activation by H_3PO_4 in enhancing the dye uptake of BP-AC (Figure 5c).

Furthermore, investigating the effects of H_3PO_4 impregnation ratios on BP-AC fabrication was deemed necessary. The utilization of H_3PO_4 serves dual functions: facilitating the pyrolytic breakdown of the initial substance and establishing a cross-linked framework. Moreover, H_3PO_4 facilitates the creation of both micropores and mesopores within the resultant activated carbon, and these pores assume a pivotal role in the adsorption of dyes by regulating the accessibility of adsorbate molecules [21]. Numerous research investigations have highlighted that the impregnation ratio is the primary factor influencing the enhancement of porosity and surface area in AC originating from lignocellulosic sources. Kumar and Jena [22] conducted a study focusing on the influence of H_3PO_4 impregnation ratios during the production of activated carbon from Fox nut shell. The authors found a substantial increase in the AC's BET surface areas and total pore volumes as the impregnation ratio increased from 0.5 to 1.5. A similar pattern was observed in AC derived from castor seed hull, where a maximum surface area was achieved at an impregnation ratio of 0.80 [23].

Among the three investigated dyes, ACH4 exhibited the highest removal efficiency, leading to the selection of an H_3PO_4 impregnation ratio of 1:2 for further investigation. Notably, this adsorbent demonstrated relatively high adsorption capacities (98.64, 95.53, and 96.03 mg g^{-1} for RhB, MO, and MR, respectively) compared to other reported ACs (Figure 5d). Such results were in harmony with several reported studies. For example, Jawad et al. [24] employed a microwave heating process to convert rubber seed pericarp (RSP) biomass waste into activated carbon (AC), utilizing H_3PO_4 as the activating agent for subsequent investigation of its adsorption capacity for Methylene Blue (MB). Their findings indicated that the optimal conditions for preparing RSP-derived AC involved an impregnation ratio 1:2 (RSP: H_3PO_4).

In this study, using H_3PO_4 as an activation agent for BP-AC fabrication proved more effective than KOH. This finding is consistent with numerous research reported in the literature. Brito *et al.* [25] synthesized ACs from yellow mombin fruit stones, utilizing a chemical impregnation ratio 1:1 of H_3PO_4 and KOH. They concluded that the use of H_3PO_4 had a more pronounced impact on the precursor's structure, resulting in an adsorbent with a larger surface area and increased pore size, which enhanced its ability to adsorb Dianix® royal blue CC compared to KOH. Mbarki *et al.* [26] produced ACs from Corn stigmata fibers and chemically activated them using H_3PO_4, $ZnCl_2$, and KOH, all with a chemical impregnation ratio of 1:2, subsequently evaluating their MB adsorption efficiencies. Their findings indicated that AC-H_3PO_4 exhibited significantly higher MB removal (99.09%) compared to AC-$ZnCl_2$ (26.22%) and AC-KOH (43.47%). This difference could be attributed to the greater specific surface area of carbons activated with H_3PO_4 than those prepared using $ZnCl_2$ and KOH.

Kinetic study

With high dye adsorption performance, ACH4 was selected for kinetic study. This study was conducted by using four models (pseudo-first-order (PFO), pseudo-second-order (PSO), Elovich, and Bangham [8]. As can be seen from Figure 6, almost all models fitted with data points, except for the PFO model. Moreover, the R^2 values of PSO were the highest at 0.9998, 0.9995, and 0.9981 for all models of RhB, MO, and MR adsorption, respectively, onto the ACH4. This finding suggests that the chemisorption process may control the kinetic energy, in which functional groups play a vital role in the adsorption. Several studies have indicated that PSO was the most appropriate model when describing the kinetic of crystal violet dye onto Ni ferrite [27], RhB dye onto minerals [28], and stalk corn activated carbon [29].

CONCLUSIONS

Two chemical agents, H_3PO_4 and KOH, were applied to Bidens pilosa L. to derive a range of activated carbons. The findings revealed that the carbon activated with H_3PO_4 exhibited superior efficacy in adsorbing RhB, MO, and MR compared to the carbon activated with KOH. Specifically, the carbon activated with a 1:2 impregnated ratio of H_3PO_4 (referred to as ACH4) demonstrated the highest adsorption capacity for dye removal. Notably, this adsorbent

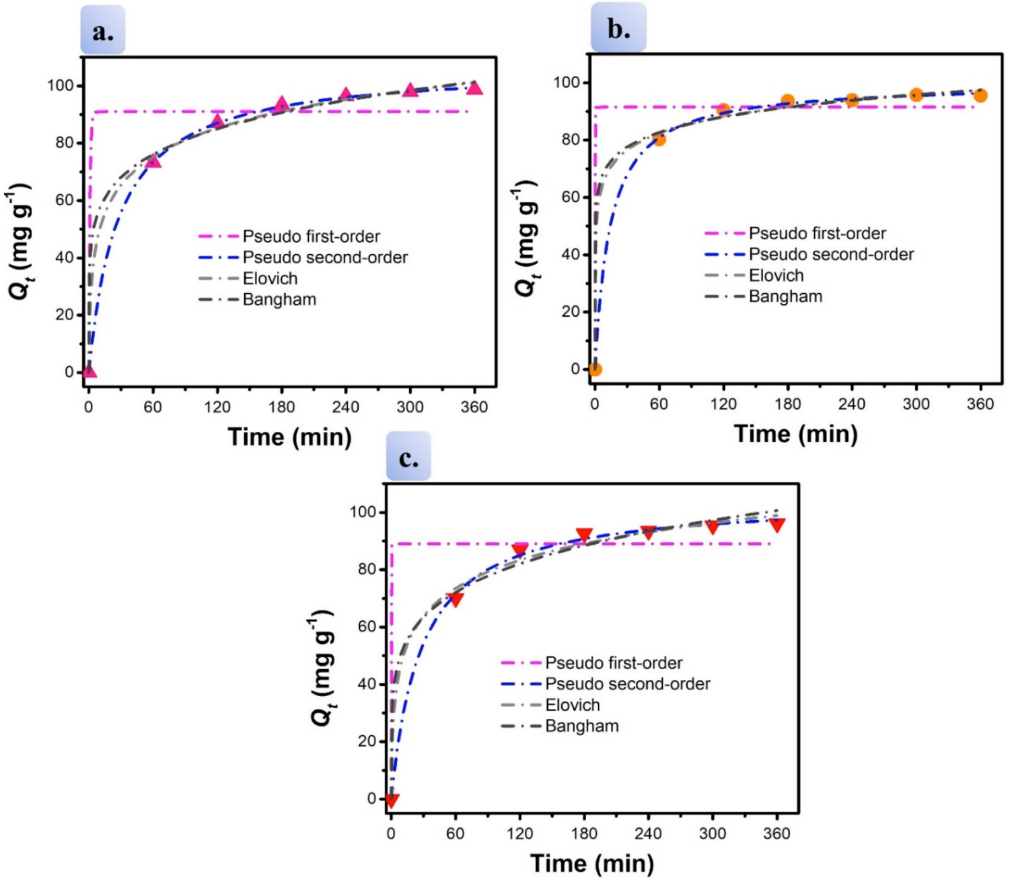

Figure 6. Kinetic models of adsorption of RhB, MO, and MR, respectively onto the ACH4.

displayed relatively elevated adsorption capacities (98.64, 95.53, and 96.03 mg g^{-1} for RhB, MO, and MR, respectively) in comparison to previously reported biomass-derived activated carbons. FTIR analysis revealed the prevalence of acidic surface functional groups, facilitating adsorption. The SEM imagery illustrated a progressive formation of pores resulting removing volatile substances and impurities. Consequently, the carbons investigated in this study hold promise for application in the removal of organic dyes from aqueous environments and stand to significantly mitigate environmental impacts related to water pollution from dyes, and the reduction of discarded biomass residues of *Bidens pilosa* weed.

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