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ORIGINAL RESEARCH ARTICLE

Utilization of agro-waste derived activated carbon for wastewater treatment: Adsorptive removal of brilliant blue dye

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ABSTRACT

Adsorption remains one of the most effective and widely applied methods for removing pollutants from water and wastewater. However, the high cost of conventional industrial adsorbents limits their application, particularly in developing countries. To address this challenge, the present study focuses on creating a low-cost, eco-friendly biosorbent derived from pomegranate peel waste. The biomass was converted into activated carbon (PPAC) through a microwave-induced chemical activation method using phosphoric acid (H_3PO_4) as the activating agent. The preparation involved impregnating dried and pulverized pomegranate peels with concentrated H_3PO_4, followed by microwave irradiation to induce carbonization and activation. The resultant activated carbon was thoroughly washed to neutral pH and dried before use. The surface morphology and physicochemical properties of the PPAC were characterized using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM). These analyses confirmed the presence of abundant functional groups (e.g., –OH, –COOH, and $–PO_4$), an amorphous carbon structure, and a highly porous surface, all of which contribute to enhanced adsorption capacity. The adsorptive performance of PPAC was evaluated in batch experiments for removing Brilliant Blue (BB), a synthetic cationic dye, from aqueous solution. Several operational parameters were optimized, including pH, initial dye concentration, contact time, and temperature. The results revealed maximum dye removal efficiency (89.36%) at pH 6. Furthermore, under optimized conditions—pH 6, adsorbent dose of 0.05 g/100 mL, BB concentration of 60 mg/L, and contact time of 60 minutes—PPAC demonstrated an impressive removal efficiency of up to 89 %.These findings confirm the potential of pomegranate peel-derived activated carbon as a sustainable and cost-effective biosorbent for treating dye-contaminated wastewater, particularly in resource-limited settings.

Keywords: Adsorption; dye; brilliant blue (bb); activated carbon; pomegranate peel

1. Introduction

Dealing with synthetic dyes from industrial sectors, such as textiles, printing, plastics, and cosmetics, into water bodies poses a serious environmental and public health challenge. These dyes are often toxic, non-biodegradable, and resistant to conventional treatment, making their removal from wastewater essential to prevent long-term environmental damage and bioaccumulation in the food chain [1]. Among various treatment technologies, adsorption has gained widespread acceptance as an effective method due to its simplicity, cost-effectiveness, and high efficiency in removing dye contaminants even at low concentrations. However, the high cost of commercial adsorbents, such as activated carbon, limits their widespread use, particularly in developing regions. Increasing attention has turned to agricultural and food processing waste as sustainable and low-cost raw materials for adsorbents to address this limitation. Among these materials, pomegranate peel (Punica granatum), an abundant agricultural waste, offers tremendous potential due to its rich lignocellulose content, its natural functional groups (such as hydroxyl, carbonyl, and carboxyl groups), and its porous structure that facilitates adsorption processes[2-4].

Dyes are classified as colored substances that adhere to textiles, paper, and biological tissues to impart color. They are widely used in many industries, including textiles, food, cosmetics, and pharmaceuticals[1, 5]. Dyes are typically classified based on their chemical structure (e.g., azo, anthraquinone, and triphenylmethane) or their method of application (e.g., acid, reactive, and basic). Dyes used in industrial applications are often synthetic and can pose significant environmental concerns due to their persistence, resistance to degradation, and potential toxicity. Wastewater from dyeing processes is an essential source of water pollution, as many dyes are resistant and difficult to remove using conventional treatment methods [6-10].

Brilliant Blue (BB), also known as Brilliant Blue FCF (E133), is an anionic synthetic dye of the triphenylmethane class with a molecular weight of approximately 792.85 g/mol. It is highly soluble in water and contains sulfonate groups that impart a negative charge under neutral conditions, strongly influencing its interaction with adsorbent surfaces. Due to its high stability and resistance to biodegradation, BB persists in aquatic environments and can pose ecological risks. Consequently, its removal from wastewater, particularly from textile and food industries, is often investigated using adsorption-based approaches.[11-14].

Recent studies have demonstrated that chemical activation with phosphoric acid (H_3PO_4) coupled with microwave-catalyzed carbonization can significantly enhance the surface area, porosity, and surface functionality of activated carbon extracted from pomegranate peel. This preparation method enables the development of an effective bioadsorbent capable of adsorbing various types of dyes, including cationic, anionic, and azo dyes, from aqueous solutions. In addition to adsorption, other commonly used methods for dye removal include coagulation and precipitation, membrane filtration, advanced oxidation processes (AOPs), ion exchange, and biological treatments. While each method has advantages, adsorption remains the most successful due to its operational simplicity, reusability, and low production of secondary pollutants. Thus, using waste-derived activated carbon supports effective wastewater treatment and promotes waste valorization and the principles of a circular economy. Integrating these environmentally friendly methods aligns with global efforts to develop sustainable, economically and ecologically viable water purification technologies.

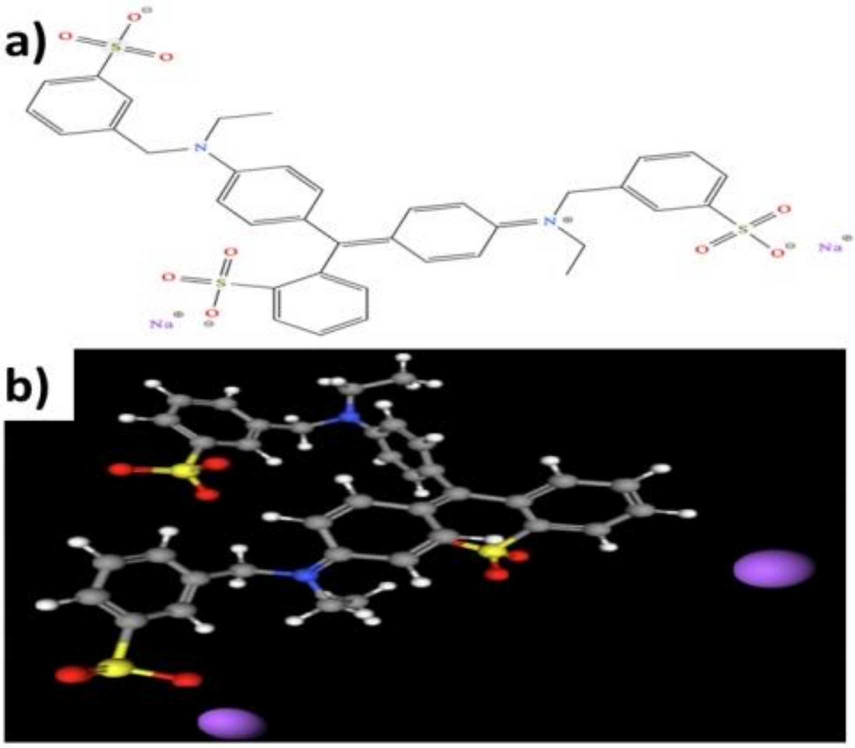

Figure 1. Chemical structure of Brilliant Blue (BB) dye: a) 2D view, b) 3D view

2. Experimental part

2.1. Preparation of phosphoric acid-activated carbon from pomegranate peel

Pomegranate peels were collected, thoroughly washed with distilled water to remove adhering dirt and soluble impurities, and then air-dried for 48 h, followed by oven drying at 80 °C for 24 h to eliminate residual moisture. The dried biomass was subsequently ground into a fine powder using a mechanical grinder and sieved to obtain a uniform particle size (<1 mm).For chemical activation, 100 g of the powdered pomegranate peel was impregnated with a 1:1 weight ratio of 85% orthophosphoric acid (H_3PO_4). The impregnation was performed by gently mixing the biomass with the H_3PO_4 solution under continuous stirring for 2 h at room temperature to ensure complete penetration of the acid into the lignocellulosic matrix. The acid-treated material was then left to soak overnight (≈12 h) to enhance activation efficiency. Following impregnation, the sample was subjected to microwave-assisted carbonization. The impregnated biomass was placed in a quartz crucible and exposed to microwave irradiation at 600 W for 10–15 min in a domestic microwave oven under limited air supply to facilitate carbonization. Microwave activation was performed at a frequency of 2.45 GHz, and the irradiation time was optimized to 10–15 min based on preliminary trials to achieve maximum surface development without structural collapse. After cooling to room temperature, the carbonized product was repeatedly washed with hot distilled water until the pH of the filtrate reached neutral (6.5–7.0), indicating the removal of residual phosphoric acid and soluble byproducts. The final product was dried in an oven at 105 °C for 12 h to obtain phosphoric acid-activated pomegranate peel carbon (PPAC). The activated carbon was then stored in airtight containers for further characterization and adsorption experiments.

2.2. Adsorption study

A stock solution of BB dye was diluted to obtain standard solutions with concentrations ranging from 10 to 100 mg/L. For the batch adsorption experiments, 100 mL conical flasks were each filled with 0.1 g of adsorbent and 100 mL of dye solution, adjusted to an initial pH of 7. During the batch adsorption experiments, the solutions were agitated using a mechanical orbital shaker operated at 120 rpm to ensure uniform mixing and effective contact between the adsorbent and the dye solution. The initial pH of the solutions was adjusted to 7 using 0.1 M HCl or 0.1 M NaOH as required, and the pH was monitored throughout the experiments using a calibrated digital pH meter to ensure stability during the adsorption process. Following agitation, the suspensions were centrifuged to separate the adsorbent from the solution, and the residual dye concentration in the supernatant was determined spectrophotometrically at 630 nm. To evaluate the effect of adsorbent dosage, experiments were conducted at a fixed dye concentration of 70 mg/L, using PPAC dosages ranging from 0.01 to 0.09 g per 100 mL. Additionally, the influence of initial dye concentration on removal efficiency was examined in the range of 10–100 mg/L, while maintaining a constant adsorbent dosage of 0.05 g per 100 mL. The equilibrium adsorption capacity was then calculated using the standard adsorption equation.

$$E\,\% = \frac{C_o - C_e}{C_o} \times 100 \tag{1}$$

$$Qe = \frac{(C_o - C_e)V}{m(g)} \tag{2}$$

where C_o (mg/L) is the initial concentration of dye, Ce (mg/L) is the concentration of BB dye at equilibrium, V (mL) is the volume of the solution, and m (g) is the weight of the PPAC.

3. Result and discussion

3.1. Characterization for adsorbent/adsorbate

Scanning electron microscopy (SEM) analysis was performed to investigate and confirm the morphology of the prepared surfaces, the elemental properties of the nanostructures, and the porous structure. Figure 4 shows the SEM analysis. (a)Pomegranate peels PP, (b); AC; (c) PPAC; (d) PPAC after adsorption of BB dye. AC was found to have a smooth and homogeneous surface without any irregularities (Figure 2a). Figure 2b shows that the surface morphology appears smooth with visible cavities after activation[15]. The smoothness of PPAC is a vital parameter that affects the increase in the adsorption capacity because it increases the surface area. The added acid activation is a molecule that can easily increase the activity at the active site. Thus, in Figure 2,c, d, the surface morphology of PPAC after adsorption was rough, irregular, and heterogeneous, well-distributed across the nanomaterial due to the H3PO4 loading on the surface, indicating the successful adsorption process[16, 17].

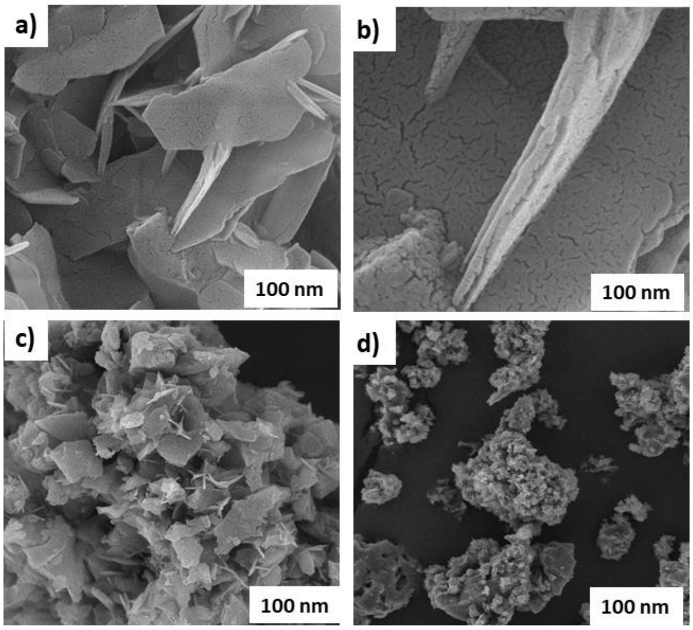

Figure 2. FESEM image of a)Pomegranate peels PP , b) AC , c) PPAC , d) PPAC after adsorption

The transmission electron microscope (TEM) image shown in Figure 3 shows unactivated pomegranate peels (AC) and those activated with PPAC, which is incorporated into the activated carbon (PPAC). PPAC appears as regular spheres with irregular black shapes and tends to form chain-like clusters at 200 nm wavelengths. Furthermore, the surface of PPAC is covered with a transparent layer, in which the acid incorporated into the activated carbon is observed. This layer is pivotal in improving stability and increasing the surface area, making it an essential component in manufacturing environmentally friendly activated carbon[18, 19].

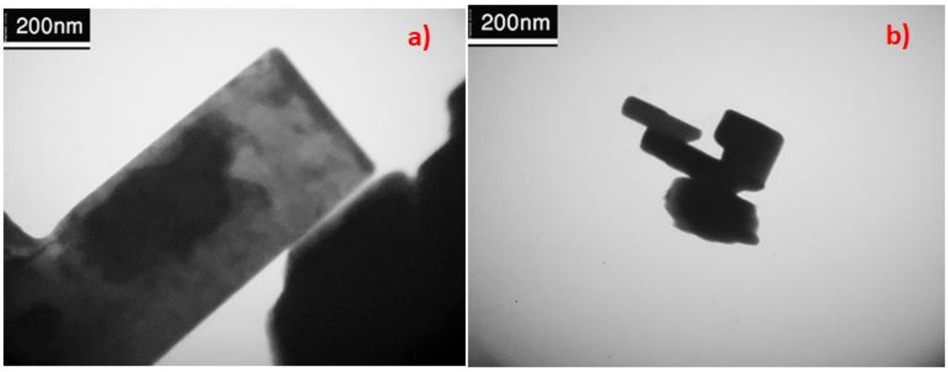

Figure 3. The transmission electron microscope (TEM) image of a) AC, b) PPAC

The X-ray diffraction (XRD) analysis of the phosphoric acid–activated carbon derived from pomegranate peel was conducted to investigate its crystalline or amorphous nature. The XRD pattern revealed a broad diffraction peak centered around 2θ ≈ 23°–25°, characteristic of amorphous carbon[20]. This broad halo indicates the presence of disordered carbon structures with low graphitization, typical of activated carbon materials obtained from lignocellulosic precursors. Additionally, the absence of sharp, well-defined peaks suggests that the activation process with H_3PO_4 disrupted the crystalline regions of the biomass, promoting the formation of a highly porous and irregular structure. The lack of crystalline impurities such as silica or metal oxides confirms the purity and effectiveness of the chemical activation. Incorporating phosphoric acid likely facilitated crosslinking reactions and the development of micro- and mesopores, contributing to the disordered arrangement of carbon atoms. This amorphous structure is advantageous for adsorption applications, as it typically corresponds to a high surface area and abundant active sites[21, 22]. Overall, the XRD results confirm that the phosphoric acid–activated carbon from pomegranate peel possesses a predominantly amorphous structure, which is suitable for adsorption processes such as dye removal from aqueous media, as shown Figure 4

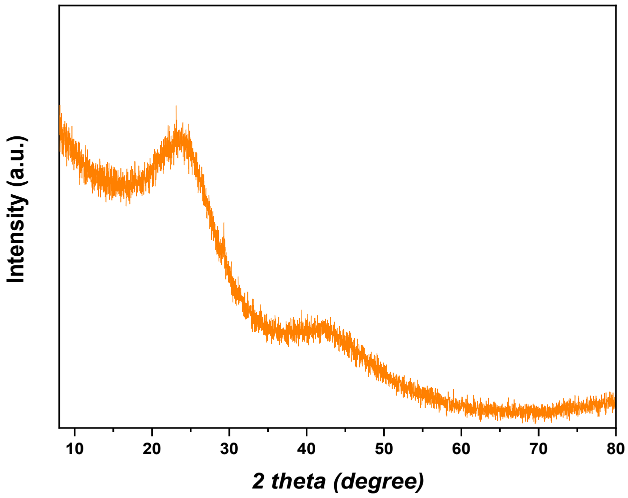

Figure 4. X-ray diffraction (XRD) analysis of PPAC surface

3.2. Effect of adsorbent dosage on brilliant blue (BB) dye removal

The impact of pomegranate peel–derived activated carbon (PPAC) dosage on the adsorption efficiency of Brilliant Blue (BB) dye was evaluated across a range of adsorbent masses (0.01–0.09 g) at a constant temperature of 30 °C, pH 7, and an initial dye concentration of 100 mg/L (as illustrated in Figure 5). The results demonstrated a significant increase in dye removal efficiency with increasing PPAC dosage. Specifically, the removal efficiency rose from 59.5% at 0.01 g to 96.9% at 0.09 g of PPAC. An intermediate dose of 0.05 g achieved 89.52% removal. This enhancement in dye removal is primarily attributed to the increased availability of active adsorption sites as the adsorbent dose increases[23]. A higher mass of PPAC introduces a greater surface area and more functional groups capable of interacting with dye molecules. Consequently, the probability of dye–adsorbent interactions is elevated, improving the overall adsorption capacity. Moreover, at higher dosages, the number of active sites surpasses the number of dye molecules present, ensuring that the adsorption sites remain primarily unsaturated during the process. However, beyond a specific dosage, the removal efficiency may plateau due to the saturation of available dye molecules in the solution, rather than a limitation of the adsorbent's capacity. These findings are consistent with prior studies that associate increased adsorbent dosage with improved pollutant removal due to the enhanced accessibility of binding sites[24, 25].

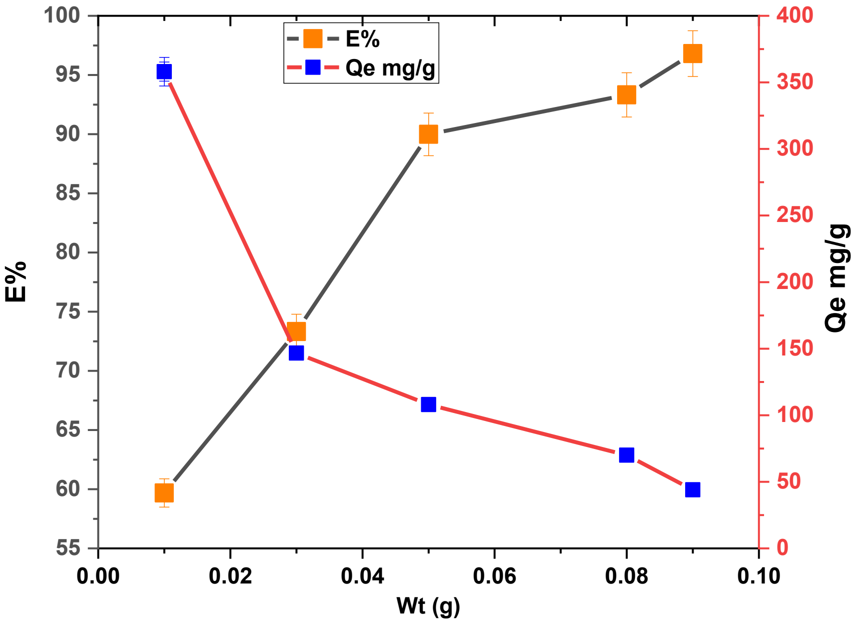

Figure 5. Effect of adsorbent dosage on Brilliant Blue (BB) Dye removal

3.3. Effect of pH

pH is a crucial factor affecting the adsorption efficiency of dye adsorbents, as it is closely related to the surface charge of the adsorbent. During the adsorption process, the pH of the solution affects the surface properties and overall efficiency. It is worth noting that the BB dye begins to lose its color when the pH exceeds 10.0, which may lead to misinterpretation of the adsorption results. Therefore, a pH range of 3.0–10.0 was chosen to study the adsorption process[26, 27]. As shown in Figure 6, the adsorption efficiency of the BB dye from the composite hydrogel increased significantly as the solution pH increased from 3.0 to 10.0. The positive charge density increased because a large amount of hydrogen ions (H+) in the solution protonate the functional groups in the composite hydrogel at low pH. Meanwhile, the composite hydrogel is easily passivated under acidic conditions, weakening the adsorption effect of the BB dye in the solution[28, 29].

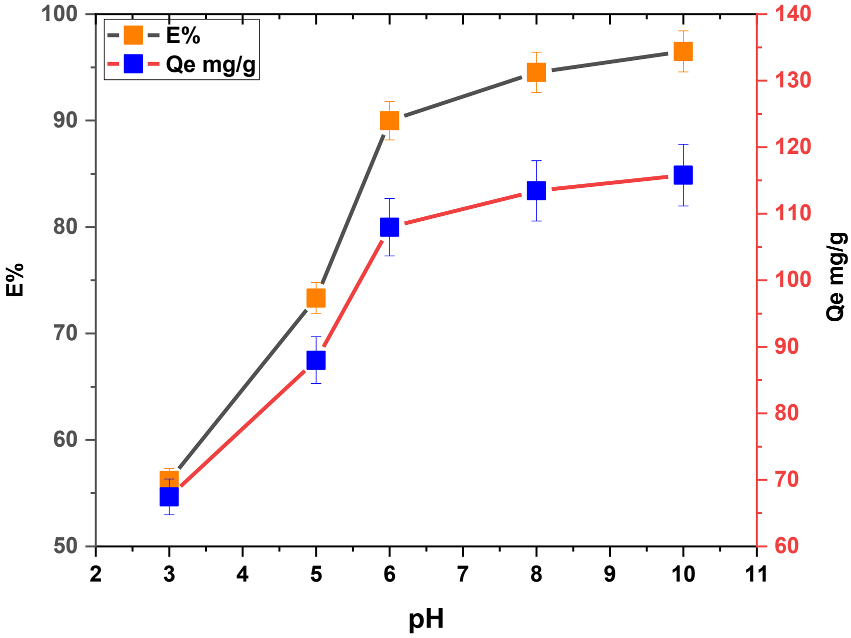

Figure 6. Effect of pH solution on the removal of BB dye

3.4. Effect of contact time

One of the most vital factors studied is the effect of equilibrium time on adsorption capacity. This process was studied over different periods ranging from 5 to 60 minutes of contact between the superabsorbent activated carbon and the dye molecules, and the results are shown in Figure 7. The adsorption rate increases until it reaches a specific limit and then reaches a constant value. Increasing contact time increases the chances of interaction between the adsorbent and the adsorbent molecules, but after one particular period of time (90 minutes for BB), the rate becomes constant due to the equilibrium between the adsorbed dye molecules and those remaining in the solution[30, 31].

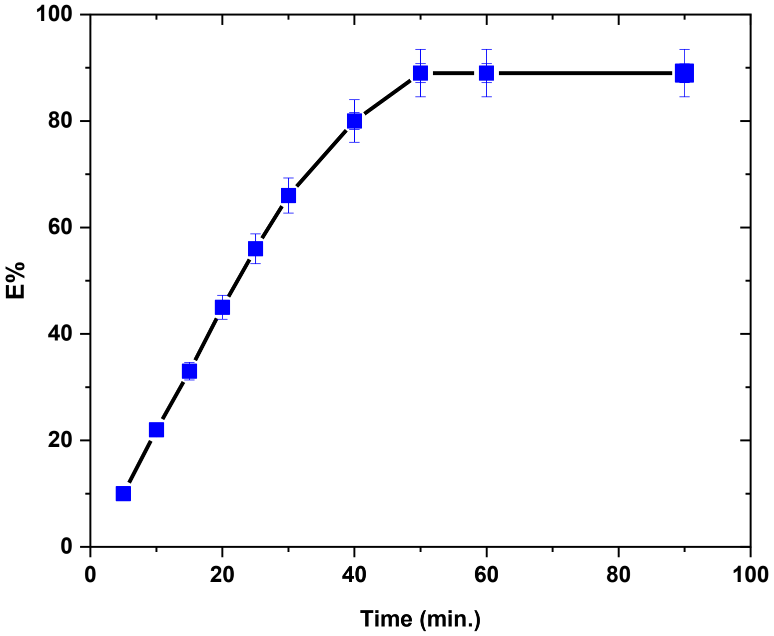

Figure 7. Effect of equilibrium time on adsorption capacity

3.5. Effect of initial concentration of BB dye on adsorption using activated carbon

The initial concentration of Brilliant Blue (BB) dye plays a crucial role in the adsorption process on activated carbon. At low concentrations, the available active sites on the activated carbon surface are sufficient to absorb most of the dye molecules, resulting in high removal efficiency. However, as the dye concentration increases, the adsorption capacity of the carbon material becomes a limiting factor. The dye molecules compete intensely for a limited number of active binding sites at higher concentrations. This competition may result in a relative decrease in the removal rate, although the absolute amount of dye adsorbed (mg/g) may increase due to the increased driving force for mass transfer between the solution and the adsorbent surface. Furthermore, higher initial concentrations enhance the concentration gradient, improving the diffusion rates of Brilliant Blue BB molecules within the porous structure of the activated carbon. However, saturation of the adsorption sites may eventually occur, causing a plateau in the adsorption capacity.

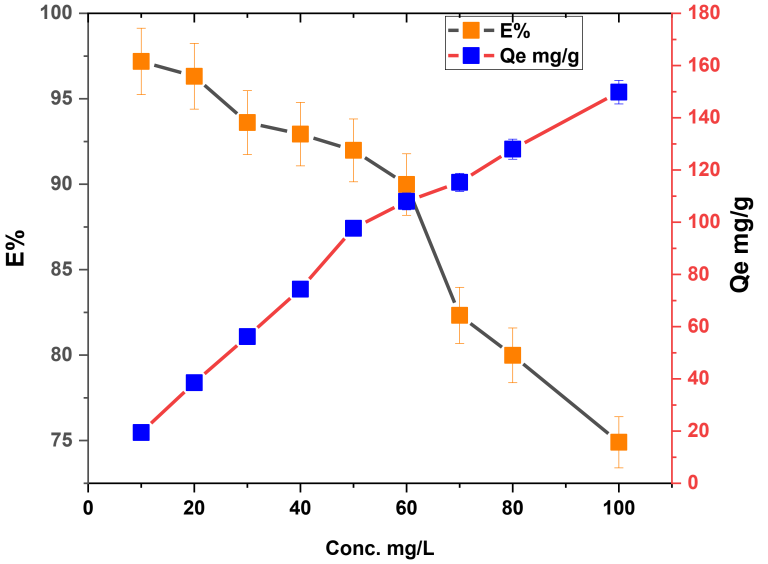

Figure 8. Effect of initial concentration of BB dye on adsorption using activated carbon

3.6. Effect of temperatures

One of the most critical factors that has a clear impact on the adsorption process, through which it is possible to determine whether the reaction is exothermic or endothermic, as well as to determine the spontaneity of the response, is the effect of dye concentration at three different temperatures: 10-40 ºC. The results are presented in Figure 9. The results show that the maximum adsorption amount is observed in the BB dye case, where the adsorption efficiency was 107.77 mg/g. In addition, the effect of temperature was found to be very significant in all cases[32, 33].

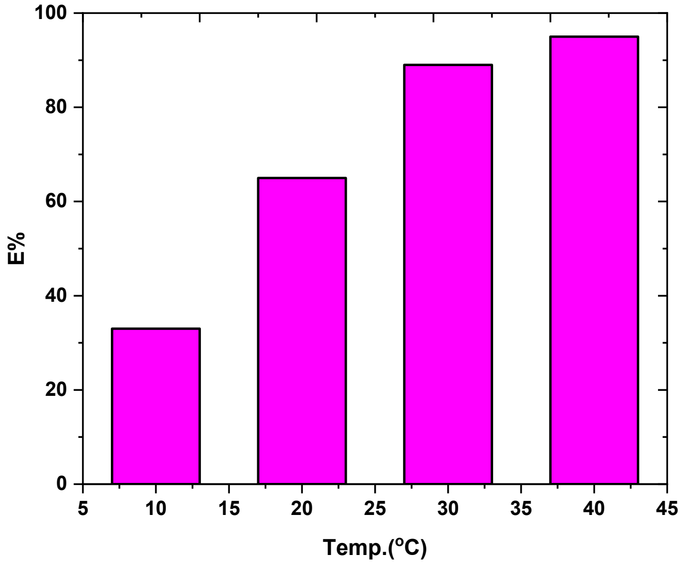

Figure 9. Effect of Temperature on the removal of BB dye

3.7. Comparison of activated carbon for the removal of BB dye with other adsorbents

The maximum adsorption capacity of the PPAC for BB dye demonstrates a clear advantage over conventional adsorbents. As shown in **Table 1**, the PPAC outperforms materials such as commercial activated carbon, which generally exhibit lower uptake values.

Table 1. Comparison of activated carbon for BB dye removal with other commercial activated carbon

Adsorbent (precursor/type)	Dye	Langmuir Qmax (mg·g^{-1})	Optimum pH	T (°C)	Key conditions (dose, time, Ci range)	Source
Your adsorbent (this study)	BB	107.77	7	25	dose 0.05 g; 5–60 min; C_0 10–100 mg·L^{-1}	In this study
Sawdust-derived **ZnO@AC** composite	MO	**44.5**	3	20	dose 1.2 g·L^{-1}; 45–60 min; C_0 10–200 mg·L^{-1}	[34]
Sawdust-derived **ZnO@AC** composite	MR	**46.0**	3	20	dose 1.2 g·L^{-1}; 45–60 min; C_0 10–200 mg·L^{-1}	[34]
Poultry-manure **biochar**	MO	**20.8**	4	(noted across 25–45)	C_0 100 mg·L^{-1}; adsorbent 2 g; Langmuir linear fit	[35]
Commercial activated carbon	MO	**129.3**	3	25	30 min contact; bench-scale batch	[36]
Rumex abyssinicus-derived **biochar**	MR	**42.34**	6	25	equilibrium ≈40 min; batch setup	[37]

4. Conclusion

In this research, we present an environmentally friendly technique for facilitating the manufacture of activated carbon from agricultural wastes to prepare multi-layered superabsorbent activated carbon based on acid activation, thereby increasing the surface active sites. Various characterization techniques revealed the successful fabrication of the superabsorbent activated carbon. Microwave-assisted phosphoric acid activation significantly enhances the adsorption capacity due to rapid and uniform heating, which promotes efficient pore development and surface functionalization, resulting in a stable porous structure with superior performance compared to conventional activation methods. The maximum adsorption capacity, as calculated, was 107.77 mg/g of BB dye. Interestingly, the maximum dye removal rate was achieved with the minimum dose of superabsorbent activated carbon. As the weight of the activated carbon increased, the removal rate increased significantly, while the removal efficiency decreased conversely, which can be attributed to its layered structure. The functional groups on the superabsorbent activated carbon were essential in the adsorption process. This study provides an alternative to the traditional, cost-effective, and efficient method.

Conflict of interest

The authors declare no conflict of interest

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