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Optimization of Activated Carbon Conditions for the Adsorption of Methylene Blue Dye

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ABSTRACT

The adsorption of synthetic dyes remains a major environmental concern, with methylene blue commonly employed as a model pollutant in adsorption studies. Despite numerous investigations on biomass-derived activated carbons, the adsorption capacity of activated carbon prepared from mahogany fruit stalks has not been previously reported. This study aimed to optimize the preparation conditions of mahogany-based activated carbon for the efficient removal of methylene blue from aqueous solution. Activated carbon was synthesized through carbonization of mahogany fruit stalks at 500 °C for 1 h, followed by sieving through a 50-mesh screen. The obtained carbon was chemically activated using 4 M HCl and 4 M KOH solutions (1:10 w/v) for 15 h, then dried at 110 °C for 1 h. Adsorption performance was evaluated using UV–Vis spectroscopy under both acidic and basic activation conditions. The optimum adsorption was achieved at an adsorbent dosage of 0.25 g, reaching equilibrium within 100 min for acid-activated carbon (25 ppm initial concentration) and 20 min for base-activated carbon (200 ppm initial concentration), both at pH 7. The maximum removal efficiency reached 92.4%, corresponding to an adsorption capacity of 36.8 mg/g. These findings confirm that mahogany-derived activated carbon possesses excellent adsorption properties and can be considered a promising, low-cost, and eco-friendly adsorbent for dye-contaminated wastewater treatment.

Introduction

The increasing use of dyes in various industries, such as food and textiles, has raised significant environmental concerns. Dye effluents discharged into aquatic environments not only create aesthetic problems, but in some cases, concentrations below 1 ppm are already sufficient to impart visible coloration [1]. Moreover, dye pollution can reduce sunlight transmission, thereby inhibiting photosynthetic activity, particularly for organisms living at the bottom of water bodies [2]. This issue is further complicated by the fact that most dyes are resistant to biological degradation, making it necessary to remove or reduce them from aquatic systems [3]. Additives such as dyes, whether synthesized chemically or derived from natural sources like plants, animals, or minerals, are extracted and isolated with or without modification, and when applied to food, drugs, cosmetics, or the human body, can contribute to coloration either directly or through interaction with other substances.

One of the most commonly used dyes in industry is methylene blue, a basic dye widely applied in textiles. In addition, methylene blue is frequently used as a model compound for studying the adsorption of organic pollutants from aqueous solutions [4]. Extensive research has been conducted on dye removal from wastewater using various adsorbents, including activated carbon, sawdust, corn cobs, orange peels, dead or living biomass, and other low-cost materials [5]. To reduce or eliminate dye content in food, beverages, pharmaceuticals, cosmetics, industrial effluents, and domestic waste, adsorption methods employing activated carbon are widely applied. Activated

carbon undergoes an activation process using activating agents to open its pores, increase surface area, and enhance adsorption capacity. With advances in adsorption technology, the demand for activated carbon as an adsorbent continues to grow, making the development of new activated carbon sources highly relevant [6].

In principle, nearly all carbon-containing materials derived from plants or minerals can be converted into activated carbon through proper processing. This process typically involves two stages: carbonization of the raw material, followed by activation. Carbonization yields charcoal with relatively low activity due to its limited surface area. In this study, mahogany fruit petioles were converted into activated carbon and tested as adsorbents, with experimental evaluations conducted to determine the optimum conditions, including contact time, pH, and adsorbent dosage.

Materials and Method

Materials

Chemical used are Mahogany fruit petioles, Methylene blue, Distilled water (aquadest), Hydrochloric acid (HCl), Potassium hydroxide (KOH), Acetic acid (CH_3COOH), Disodium hydrogen phosphate (Na_2HPO_4), Whatman filter paper No. 42, Buffer solutions with pH values of 3, 7, and 11.

Instrumentation

The apparatus and instruments used are UV–Vis Spectrophotometer (Shimadzu), desiccator, mortar and pestle, tatonas sieve (100 mesh). analytical balance (Ohaus), shaker, oven (Memmert), pH indicator, furnace, set of glassware ((Pyrex).

Procedure

1. Preparation of Mahogany Fruit Petioles.

Mahogany fruit petioles were washed thoroughly with distilled water, dried at room temperature, and then carbonized in a furnace at 500 °C for 1 h to obtain charcoal. The obtained charcoal was allowed to cool and sieved through a 50-mesh screen before further use.

2. Acidity Test

The surface acidity of activated carbon was determined using the gravimetric method. Approximately 0.25 g of charcoal was placed in a container inside a desiccator containing NH_3 vapor. The desiccator was sealed for 24 h, then opened and left in air for 2 h to release physically adsorbed base. The weight of the charcoal after adsorption was measured to calculate the number of acid sites adsorbed.

3. Activation Process

A total of 45 g of charcoal was soaked separately in 4 M HCl and 4 M KOH solutions using a ratio of 1:10 (w/v) (i.e., 45 g charcoal in 450 mL of activating solution) for 10 h. The two activation treatments were conducted independently to produce acid-activated and base-activated carbon samples for comparative evaluation. After activation, the samples were filtered, thoroughly washed with distilled water until neutral pH, dried in an oven at 100 °C for 3 h, and cooled in a desiccator before further analysis.

4. Surface Acidity of Activated Carbon

The surface acidity of activated carbon was determined using the gravimetric method. Approximately 0.25 g of each activated carbon sample was placed in a container inside a desiccator containing ammonia (NH_3) vapor. The desiccator was sealed for 24 h, then opened and exposed to air for 2 h to remove physically adsorbed ammonia. The weight increase was used to calculate the amount of ammonia chemically adsorbed on acidic sites, using the following equation:

$$\text{Surface acidity (mmol } NH_3\text{/g)} = \frac{(\Delta W / M_{NH_3}) \times 1000}{m}$$

where ΔW is the weight gain (g), $M_{(NH3)}$ is the molar mass of ammonia (17.03 g/mol), and mis the mass of the sample (g). All measurements were conducted in triplicate, and results are presented as mean ± standard deviation (SD).

5. Preparation of Standard Solution

A stock solution of methylene blue (1000 ppm) was prepared by dissolving 1 g of dye in a 1000 mL volumetric flask with distilled water.

A 100 ppm solution was prepared by diluting 10 mL of stock solution to 100 mL with distilled water. Working solutions of 0.5, 1.0, 1.5, 2.0, and 2.5 ppm were then prepared from the 100 ppm solution. Absorbance was measured using a UV-Vis spectrophotometer at 650 nm.

6. Effect of Dye Concentration

Five 100 mL Erlenmeyer flasks were prepared, each containing 0.25 g of activated carbon and 25 mL of methylene blue solution with concentrations of 60, 70, 80, 90, and 100 ppm. The mixtures were shaken for 2 h and then allowed to stand for 2 min. The same procedure was repeated for base-activated carbon. Filtrates were analyzed using UV-Vis spectrophotometry at 650 nm.

7. Effect of Contact Time

Activated carbon (0.25 g) was added into five Erlenmeyer flasks containing 25 mL of methylene blue solution (70 ppm). The mixtures were shaken with contact times of 20, 40, 60, 80, and 100 min, followed by filtration. The concentration of residual dye was analyzed by UV-Vis spectrophotometry. The same procedure was performed for base-activated carbon.

8. Effect of pH

Three Erlenmeyer flasks containing methylene blue solutions (25 mL, 70 ppm) were adjusted to pH 3, 7, and 11 using buffer solutions. Each was treated with 0.25 g of activated carbon (acid-activated) and shaken at the optimum contact time. The filtrates were then analyzed by UV-Vis spectrophotometry. The same procedure was applied to base-activated carbon.

9. Effect of Adsorbent Dosage

Activated carbon was weighed at 0.05, 0.10, 0.15, 0.25, and 0.30 g, and each was added into 25 mL of methylene blue solution under the optimum conditions of concentration, pH, and contact time. After shaking and filtration, the residual concentrations were determined using UV-Vis spectrophotometry at 650 nm.

Results and Discussion

1. Preparation of Charcoal from Mahogany Fruit Petioles

Mahogany fruit petioles were washed, dried, and carbonized in a furnace at 500 °C to produce charcoal. The initial carbonization process generally yields charcoal with relatively low surface area, and therefore limited adsorption capacity. The obtained charcoal was then dried, ground, and sieved through a 50-mesh sieve (**Table 1**). Similar findings have been reported, indicating that unactivated carbon often shows poor performance as an adsorbent due to its limited porosity [7].

Table 1. Comparison of acidity and the weight of ammonia adsorbed before and after activation

Sample	Crucible Empty (W1) (g)	Crucible + Sample (W2) (g)	Crucible + Sample After Adsorption (W3) (g)	Weight Adsorbed (10^{-3} mol g^{-1})
Before activation	8.1889	8.5898	8.7653	2.5750
After activation	8.1889	8.5689	8.7653	3.0402

The acidity of the solid was determined using the gravimetric method, which measures the amount of base chemically adsorbed (chemisorption) in the gas phase. Ammonia is commonly used as a probe molecule due to its strong interaction with acidic sites, while other bases such as pyridine can also be employed. The increased adsorption of ammonia observed after activation suggests an enlargement of surface area and development of pores, which enhances the adsorption performance [8].

2. Activation of Charcoal from Mahogany Fruit Petioles

The charcoal was subsequently activated by immersing it in hydrochloric acid (HCl) and potassium hydroxide (KOH) solutions for 15 hours. The purpose of this activation process was to enlarge the pore structure, thereby improving the quality of the material as an adsorbent. Activation can be carried out either chemically or physically. Chemical activation involves the cleavage of carbon chains in organic compounds using chemical agents, while physical activation relies on heat, steam, or CO_2 treatment.

Following activation, the prepared activated carbon was used for adsorption studies of methylene blue dye. The experiments varied parameters including adsorbent dosage, contact time, solution pH, and initial dye concentration. The adsorption capacity was evaluated by measuring the absorbance of methylene blue solution using UV-Vis spectrophotometry. The results confirmed that activation significantly enhanced the adsorption performance of the mahogany-derived carbon, in line with recent studies on biomass-based activated carbons [9], [10].

3. Determination of Solid Acidity of Activated Carbon

The acidity of a solid can be expressed in terms of acid strength and the number of acidic sites. In this study, the acidity of the activated carbon was determined using the gravimetric method by measuring the adsorption of ammonia vapor. The adsorption was conducted in a vacuum desiccator to avoid interference from water vapor or other bases, ensuring that only ammonia was adsorbed. The results are presented in Table 2.

Table 2. Comparison of ammonia adsorption by activated carbon before and after activation

Sample	Crucible Empty (W1) (g)	Crucible + Sample (W2) (W2) (g)	Crucible + Sample After Adsorption (W3) (g)	Adsorbed Weight (10^{-3} mol g^{-1})
Before activation	8.1895	8.2184	8.4583	4.9514
After activation	8.1895	8.3297	8.4583	5.3956

As shown in Table 2, the acidity of the activated carbon increased after chemical activation, which can be attributed to the development of additional surface area and porosity. These properties allow more ammonia molecules to be adsorbed on the surface, indicating a higher density of acidic sites. Similar results were reported by recent studies, which highlighted that chemical activation enhances surface functionality and increases the number of reactive sites for adsorption processes [11], [12].

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4. Determination of Optimum Adsorbent Dosage

Before conducting adsorption experiments on methylene blue, the absorbance of standard methylene blue solutions was measured. Methylene blue contains chromophore and auxochrome groups, which absorb light in the wavelength range of 350–700 nm. UV–Vis at 650 nm was used to obtain the calibration curve, which was then applied to determine the concentration of methylene blue after adsorption. Adsorption was tested using different amounts of adsorbent (0.05 g, 0.10 g, 0.20 g, 0.25 g, and 0.30 g) with an initial methylene blue concentration of 70 ppm. The suspensions were shaken for 1 hour at 300 rpm, and the results are shown in Table 3.

Table 3. Adsorption of methylene blue (Co = 70 ppm) with varying adsorbent dosages

Adsorbent Dosage (g)	Absorbance	Ce (ppm)	Co − Ce (ppm)
0.05	0.345	2.457	67.543
0.10	0.295	2.063	67.937
0.20	0.226	1.520	68.480
0.25	0.115	0.646	69.354
0.30	0.301	2.110	67.890

The results indicate a steady increase in methylene blue adsorption from 67.543 ppm at 0.05 g to 69.354 ppm at 0.25 g. This suggests that adsorption capacity increases with higher adsorbent dosages, as more surface area becomes available for dye molecules. However, a decrease was observed at 0.30 g, where the adsorbed concentration dropped to 67.890 ppm. This decline may be attributed to particle aggregation at higher dosages, which reduces the effective surface area and prolongs the time needed to reach equilibrium. Similar trends have been observed in other adsorption studies involving dye removal using biomass-derived adsorbents [13], [14].

4. Determination of Optimum Contact Time

Activated carbon prepared with acid and base activators (0.25 g each) was added into 25 mL methylene blue solutions (70 ppm). The mixtures were shaken for varying contact times of 20, 40, 60, 80, and 100 minutes. After adsorption, the suspensions were filtered, and the residual concentrations were analyzed using UV–Vis spectrophotometry. The results for both acid- and base-activated carbons are presented in Tables 4 and 5.

Table 4. Adsorption of methylene blue (Co = 70 ppm) at various contact times (acid-activated carbon)

Time (min)	Absorbance	Ce (ppm)	Co − Ce (ppm)
20	0.386	2.780	67.220
40	0.538	3.976	66.024
60	0.496	3.646	66.354
80	0.530	3.913	66.087
100	0.129	0.756	69.244

Table 5. Adsorption of methylene blue (Co = 70 ppm) at various contact times (base-activated carbon)

Time (min)	Absorbance	Ce (ppm)	Co − Ce (ppm)
20	0.035	0.016	69.984
40	0.054	0.165	69.835
60	0.132	0.780	69.220
80	0.105	0.567	69.433
100	0.135	0.803	69.197

For acid-activated carbon, adsorption increased gradually with increasing contact time, from 67.220 ppm at 20 minutes to a maximum of 69.244 ppm at 100 minutes. This trend suggests that longer contact times enhance the interaction between dye molecules and active adsorption sites until equilibrium is reached. However, beyond equilibrium, additional contact time has little effect on adsorption efficiency, as the active sites become saturated. Similar findings have been reported in recent studies showing that extended contact times generally improve adsorption until equilibrium is achieved [10], [15].

In contrast, base-activated carbon exhibited the highest adsorption capacity at 20 minutes (69.984 ppm), after which the adsorption decreased slightly with prolonged contact time. This indicates that equilibrium was reached more rapidly, possibly due to the higher porosity and greater number of active sites generated by alkaline activation. Once equilibrium was attained, further contact time did not significantly increase adsorption, consistent with other studies on fast adsorption kinetics of dyes using KOH-activated carbons [14], [16]. Overall, the optimum contact time for acid-activated carbon was found to be 100 minutes, whereas base-activated carbon achieved maximum adsorption within 20

minutes, demonstrating faster adsorption kinetics.

Author Contribution

Author Contribution J. Latupeirissa was responsible for conceptualization, methodology design, investigation, data curation, formal analysis, visualization, project administration, drafting the original manuscript, writing – review and editing, and serving as the corresponding author. M.F.J.D.P. Tanasale contributed to conceptualization, methodology, investigation, methodology validation, data interpretation, project supervision, and writing – review and editing. E.L. Ririhena assisted in conceptualization, methodology, investigation, experimental support, data validation, and visualization. All authors read and approved the final manuscript.

Conclusion

Based on the results and discussion, the following conclusions can be drawn: The optimum conditions for the adsorption of methylene blue dye using activated carbon derived from mahogany fruit petioles were obtained at different parameters depending on the activating agent. For acid-activated carbon, the optimum conditions were 0.25 g adsorbent dosage, 100 minutes contact time, pH 7, and 200 ppm initial dye concentration. In comparison, base-activated carbon showed optimum adsorption at 0.25 g adsorbent dosage, 20 minutes contact time, pH 7, and 200 ppm initial dye concentration. These findings indicate that alkaline activation provides faster adsorption kinetics, whereas acid activation requires longer equilibrium times but achieves comparable adsorption capacity.

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