Thermochemical activation of bagasse-derived carbon with HCl for methylene blue dye removal

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ABSTRACT

This study shows how to make activated carbon (AC) from sugarcane bagasse by treating it with hydrochloric acid (HCl) and using it to remove methylene blue (MB) dye from water. The bagasse was soaked in 7% HCl and carbonized at 450 °C to produce a porous carbonaceous adsorbent. The tests showed that the material had a structure with small pores, a large surface area of 473.8 m²/g, many oxygen groups, and a lot of non-crystalline carbon. Tests were conducted in batches to determine the impact of contact time on the removal efficiency of the dye. The AC demonstrated rapid adsorption, achieving 95.6% MB removal within 20 minutes. Kinetic data fitted better to the pseudo-second-order model, suggesting chemisorption as the dominant mechanism. Isotherm analysis supported favorable adsorption behavior. The results show that carbon made from HCl-activated bagasse is a good, eco-friendly, and inexpensive option for getting rid of cationic dyes in wastewater. Its promising performance highlights its potential in industrial dye remediation processes.

Keywords: Activated carbon; Sugarcane bagasse; Thermochemical activation; Methylene blue; Adsorption; Wastewater treatment.

1. INTRODUCTION

The increasing discharge of synthetic dyes from the textile, paper, and leather industries poses a significant threat to aquatic environments and public health [1]. Among these dyes, methylene blue (MB) is commonly used due to its stability and vibrant color. However, prolonged exposure to MB can lead to harmful effects such as skin irritation, eye burns, and respiratory complications [2]. Conventional wastewater treatment methods, including coagulation, membrane filtration, and advanced oxidation processes, often face limitations such as high operational costs, significant energy demands, or incomplete dye removal [3, 4]. As a result, adsorption has emerged as one of the most efficient, cost-effective, and environmentally friendly techniques for dye remediation, offering simplicity, high effectiveness, and minimal production of harmful by-products [5, 6].

Activated carbon (AC) is widely recognized as a superior adsorbent due to its large surface area, well-developed porosity, and high adsorption capacity [7, 8]. However, the commercial production of activated carbon is often expensive and resource-intensive, leading to a search for sustainable and low-cost alternatives. Agricultural residues, such as sugarcane bagasse, a fibrous byproduct of the sugar industry, provide a renewable and abundant carbon source for producing activated carbon [9-11]. Bagasse is primarily composed of cellulose, hemicellulose, and lignin, making it an ideal precursor for carbonaceous materials [12, 13]. By applying thermochemical activation, particularly with chemical agents like hydrochloric acid (HCl), the surface properties, pore structure, and functional groups of carbon can be significantly enhanced, thus improving its adsorption potential for pollutants like methylene blue.

This study proposes a sustainable method to produce activated carbon from sugarcane bagasse via HCl-assisted thermochemical activation, offering a greener alternative to KOH or $ZnCl_2$. The adsorbent's ability to remove methylene blue was evaluated, with characterization performed by FTIR, SEM, and BET. Batch experiments examined contact time, dye concentration, and dosage, while kinetic and isotherm models described adsorption behavior. Results highlight HCl-activated bagasse carbon as a low-cost, efficient adsorbent, where mesoporosity and oxygenated groups synergistically facilitate chemisorption.

2. EXPERIMENTS

2.1. Chemicals and materials

Sugarcane bagasse was collected from roadside juice vendors in Cau Giay, Hanoi, after extraction. It was washed several times with distilled water to remove contaminants, sugars, and debris, then naturally dried for 48 h and oven-dried at 80 °C for 24 h to stabilize moisture before activation. Hydrochloric acid (37%, Merck) was used as the activating agent, and methylene blue (Sigma-Aldrich, analytical grade) served as the adsorbate. All other reagents were analytical grade, with deionized water used throughout.

2.2. Fabrication of activated carbon

The sugarcane bagasse was soaked in 7% HCl and carbonized in a horizontal tube furnace at 450 °C for 2 hours, with a flow rate of argon gas at 100 cm^3/min and a heating rate of 10 °C/min, as referred to and adjustment according to the previous publication [14]. Next, the material was sieved to ensure that all of the particles were uniformly sized, not greater than 1200 μm.

2.3. Characterization of activated carbon

The morphology of activated carbon (AC) was analyzed with SEM (JSM-IT800) equipped with an X-ray detector. XRD (Bruker D8 Advance) identified crystal structures over $2\theta = 10°–70°$. BET surface area was determined by N_2 adsorption–desorption using NOVA touch 2LX/Quantachrome TouchWin. FTIR spectroscopy (Nicolet iS10, 4000–400 cm^{-1}) characterized surface functional groups.

2.4. Methylene blue adsorption on activated carbo006E

Batch adsorption experiments evaluated the effects of contact time, pH (3–11), and MB concentration (10–50 mg/L). Each test used 0.05 g activated carbon in 50 mL solution, shaken at 150 rpm for 15–75 minutes at 298 K. Post-filtration, MB was analyzed by UV-Vis, and adsorption capacity (q_e) and efficiency (%) were calculated.

$$q_t = \frac{(C_o - C_t) \times V}{m} \tag{1}$$

$$H = \frac{(C_o - C_t) \times 100}{C_o} \tag{2}$$

Where: q_t (mg g^{-1}) is adsorption capacity at time t; C_0 and C_t (mg L^{-1}) are initial and time-t MB concentrations; V (mL) is solution volume; m (mg) is adsorbent mass; H (%) removal efficiency.

3. RESULTS AND DISCUSSION

3.1. Characterization of activated carbon

The SEM image in figure 1 reveals the HCl-activated sugarcane bagasse carbon with a rough, porous surface featuring irregular cavities and channels, indicating effective activation. These pores likely formed from volatile decomposition and acid-assisted etching, creating abundant attachment sites. The microstructure shows a well-developed porous network of macro- and mesopores that facilitates dye diffusion and adsorption. Such morphology enhances methylene blue uptake, consistent with the improved performance observed experimentally.

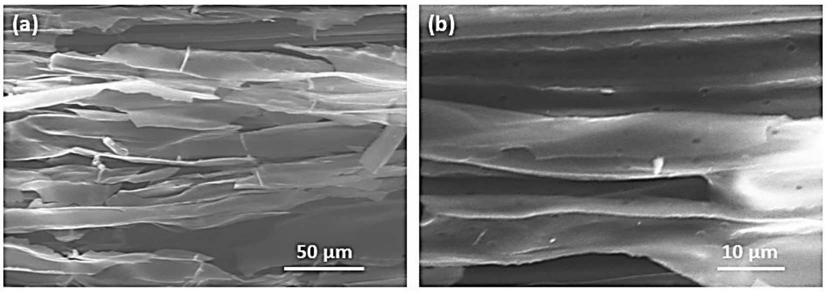


Figure 1. *SEM images of activated carbon from sugarcane bagasse.*

Figure 2a displays the XRD pattern of activated carbon obtained from sugarcane bagasse after HCl-assisted thermochemical activation. The broad diffraction peak around 2θ = 22.53° indicates the presence of amorphous carbon (PDF card 00-050-0926), typical of biomass-derived activated carbon, with limited graphitic ordering [15]. The absence of sharp crystalline peaks confirms the predominantly disordered structure, which contributes to a high surface area and favorable adsorption characteristics.

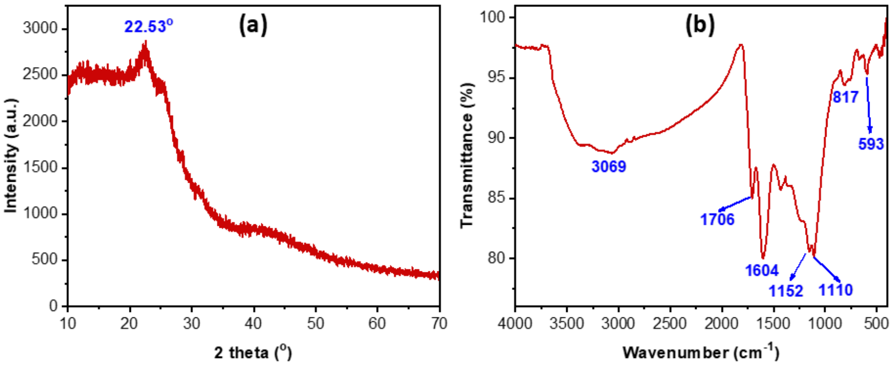


Figure 2. *XRD pattern (a) and FTIR spectroscopy (b) of activated carbon from sugarcane bagasse.*

In figure 2b, the FTIR spectrum reveals key functional groups on the carbon surface. A broad band around 3400 cm^{-1} corresponds to O–H stretching vibrations from hydroxyl groups, while peaks near 2920 cm^{-1} and 2850 cm^{-1} indicate C–H stretching [16]. The bands around 1700 cm^{-1} and 1600 cm^{-1} are attributed to C=O and aromatic C=C stretching, respectively [17]. The presence of these oxygen-containing groups enhances the material's hydrophilicity and provides active sites for methylene blue adsorption through hydrogen bonding and electrostatic interactions. Overall, both XRD and FTIR results confirm successful activation and functionalization of the bagasse-derived carbon.

Figure 3a presents the nitrogen adsorption–desorption isotherm of activated carbon derived from sugarcane bagasse, exhibiting a Type IV isotherm with a hysteresis loop, characteristic of mesoporous materials. This is consistent with the BET surface area of 473.766 m^2/g, indicating extensive porosity and a high surface area suitable for adsorption applications. The BET surface area is relatively high compared to many other bagasse-derived activated carbons, which typically range from 300–450 m^2/g depending on activation conditions [18]. The isotherm profile indicates capillary condensation in mesopores, facilitating dye diffusion. Figure 3b shows a dominant pore radius of ~1.85 nm, confirming the mesoporous structure, while a total pore volume of 0.139 cc/g supports well-developed porosity formed during HCl activation. This architecture enhances methylene blue adsorption by providing abundant active sites and enabling rapid transport of dye molecules into the adsorbent's interior. This meso/micro balance improves how quickly and how much can be absorbed by making it easier to access and interact with. Overall, the N_2 isotherm and BJH analysis confirm the effective textural development of the activated carbon.

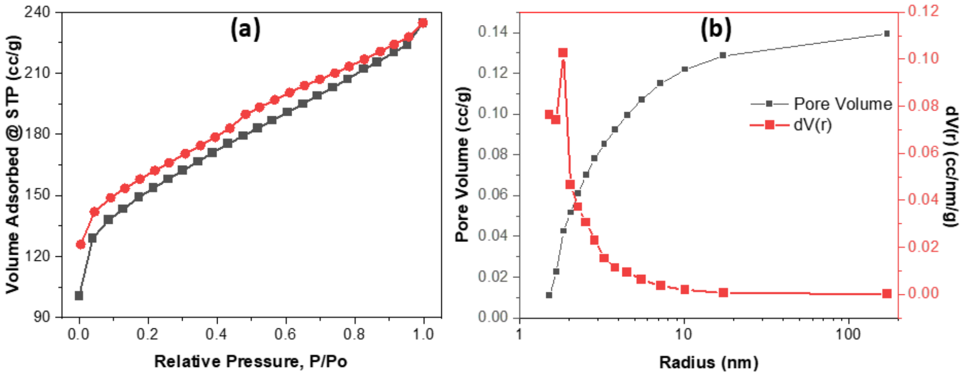
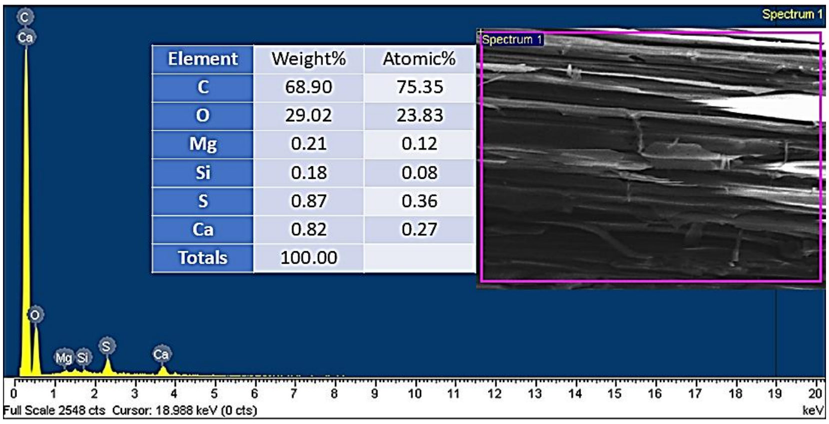


Figure 3. *Nitrogen adsorption-desorption isotherm (a) and BJH pore size distribution (b) of activated carbon from sugarcane bagasse.*

Figure 4 presents the EDX spectrum and elemental composition of activated carbon from sugarcane bagasse. Carbon dominates (68.9 wt%, 75.35 at%), confirming successful carbonization, while oxygen is also abundant (29.02 wt%, 23.83 at%), reflecting oxygenated groups such as hydroxyl, carbonyl, and carboxyl that enhance hydrophilicity and create active sites for MB adsorption via electrostatic attraction and hydrogen bonding. Trace elements, including Mg (0.21 wt%), Si (0.18 wt%), S (0.87 wt%), and Ca (0.82 wt%), likely arise from residual plant ash or activation byproducts, subtly affecting surface chemistry and ion-exchange capacity. These findings align with FTIR results, reinforcing the presence of diverse functional groups responsible for dye removal. Overall, the EDX analysis verifies a carbon-rich, functionalized material with strong potential for adsorption applications.



Element	Weight%	Atomic%
C	68.90	75.35
O	29.02	23.83
Mg	0.21	0.12
Si	0.18	0.08
S	0.87	0.36
Ca	0.82	0.27
Totals	100.00	

Figure 4. *EDX pattern and chemical composition of activated carbon from sugarcane bagasse.*

3.2. Methylene blue adsorption on activated carbon

Figure 5a shows that the methylene blue (MB) absorption band at ~664 nm decreases with increasing contact time with bagasse-derived activated carbon. The peak intensity falls rapidly within the first few minutes and is nearly quenched by 20 min, with slight blue shifts and reduced 614 nm shoulder intensity indicating weakened $\pi \rightarrow \pi^*$ transitions from π–π stacking and electrostatic interactions. Figure 5b confirms this with removal efficiency rising from 14.2% at 5 min to 95.6% at 20 min. The fast and near-complete removal reflects the material's large surface area (~474 m^2/g), mesoporous structure, and oxygenated functional groups, which together provide abundant, accessible active sites for MB adsorption.

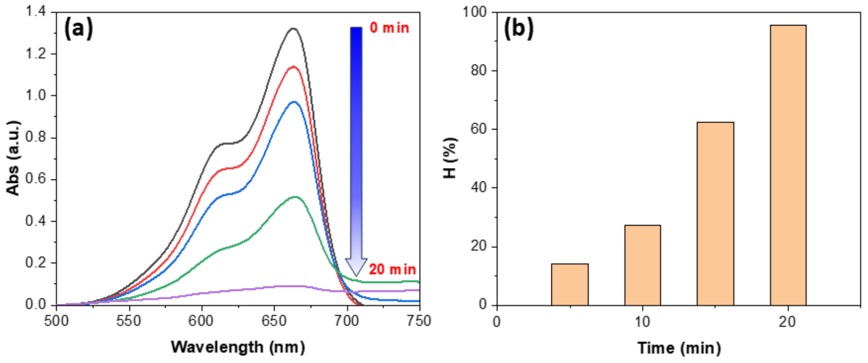
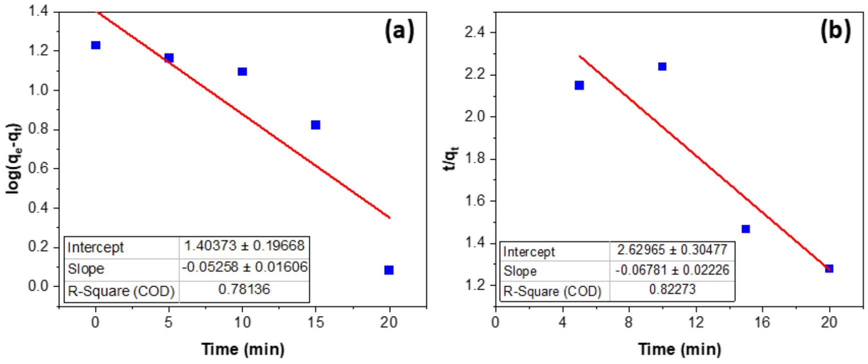


Figure 5. *UV-Vis spectroscopy (a) and removal efficiency (b) of MB by activated carbon from sugarcane bagasse.*

Figure 6 compares methylene blue adsorption onto bagasse-derived activated carbon using pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models. In the PFO model (figure 6a), the slope of 0.0526 min^{-1} gives a rate constant (k_1) of 5.26×10^{-2} min^{-1} and an equilibrium capacity ($q_{e,calc}$) of ~4.1 mg g^{-1}. However, the R^2 value of 0.781 shows only moderate agreement, suggesting that physical adsorption alone cannot explain the process. In contrast, the PSO model (figure 6b) provides a slope of 0.0678, yielding qe,calc ≈ 14.7 mg g^{-1}, which closely matches the experimental value. From a = 2.630, the PSO rate constant (k_2) is 5.6×10^{-3} g mg^{-1} min^{-1}. With a higher R^2 of 0.823, the PSO model demonstrates a better fit, indicating that chemisorption involving valence forces and electron sharing with oxygenated groups dominates the adsorption mechanism.



Intercept	1.40373 ± 0.19668
Slope	-0.05258 ± 0.01606
R-Square (COD)	0.78136

Intercept	2.62965 ± 0.30477
Slope	-0.06781 ± 0.02226
R-Square (COD)	0.82273

Figure 6. *The linear plot illustrates the pseudo-first-order (a) and pseudo-second-order (b) of MB adsorption on activated carbon from sugarcane bagasse.*

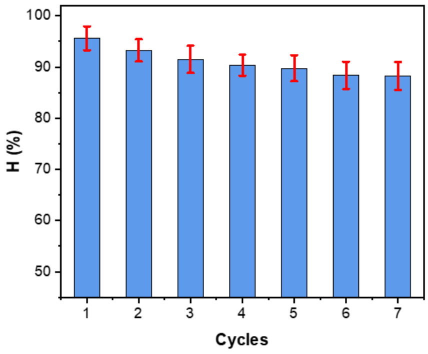


Figure 7. *The reusability of activated carbon from sugarcane bagasse as an adsorbent for the removal MB.*

Figure 7 shows the reusability of HCl-activated carbon from sugarcane bagasse for MB removal over seven cycles. Efficiency dropped slightly from 95.63% to 88.25%, indicating some site loss or incomplete desorption. Despite this decline, efficiency remained above 88%, confirming strong regeneration potential, structural stability, and reproducibility. These results emphasize the adsorbent's practicality for multiple-use wastewater treatment, offering both economic and environmental benefits.

4. CONCLUSIONS

This study demonstrated the production of activated carbon from sugarcane bagasse using hydrochloric acid, yielding a highly porous material effective for methylene blue (MB) removal. The carbon exhibited a microporous structure with a surface area of 473.8 m^2/g and abundant oxygen groups that enhanced adsorption. UV-Vis analysis showed rapid dye removal, with >95% efficiency within 20 minutes. Adsorption followed the pseudo-second-order model, indicating chemisorption driven by electrostatic attraction and π–π interactions, and isotherm results confirmed strong affinity for MB molecules. Compared to commercial activated carbons, the HCl-modified bagasse carbon is a low-cost, sustainable option for large-scale wastewater treatment. Future work should explore regeneration, multicomponent dye systems, and scale-up potential, contributing to waste valorization and environmental protection.

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TÓM TẮT

Quá trình hoạt hóa nhiệt hóa tạo carbon có nguồn gốc từ bã mía sử dụng HCl để loại bỏ thuốc nhuộm methylene blue

Nghiên cứu này mô tả cách tạo than hoạt tính (AC) từ bã mía bằng cách xử lý với axit clohydric (HCl) và ứng dụng loại bỏ thuốc nhuộm xanh methylen (MB) khỏi nước. Bã mía được ngâm trong 7% HCl và cacbon hóa ở 450 °C để tạo ra vật liệu hấp phụ carbon xốp. Các thử nghiệm cho thấy vật liệu có cấu trúc với các lỗ nhỏ, diện tích bề mặt lớn 473,8 m²/g, nhiều nhóm oxy với carbon vô định hình. Các thử nghiệm được tiến hành đánh giá ảnh hưởng của thời gian tiếp xúc, đến hiệu quả loại bỏ thuốc nhuộm. AC đã chứng minh khả năng hấp phụ nhanh, đạt được 95,6% loại bỏ MB trong vòng 20 phút ở nồng độ đầu là 10 mg/L. Dữ liệu động học phù hợp hơn với mô hình bậc hai giả, cho thấy sự hấp phụ hóa học là cơ chế chủ đạo. Phân tích đẳng nhiệt hỗ trợ hành vi hấp phụ thuận lợi. Kết quả cho thấy carbon làm từ bã mía hoạt hóa bằng HCl là một lựa chọn tốt, thân thiện với môi trường và không tốn kém để loại bỏ thuốc nhuộm cation trong nước thải. Hiệu suất đầy hứa hẹn của nó làm nổi bật tiềm năng của nó trong các quy trình xử lý thuốc nhuộm công nghiệp.

Từ khoá: Than hoạt tính; Bã mía; Hoạt hóa nhiệt hóa; Xanh methylen; Hấp phụ; Xử lý nước thải.