

Optimization of Hydrothermal Carbonization Time of *Dimocarpus longan* Peel: Adsorption Selectivity and Regeneration Performance for Dyes

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Abstract

This study investigates the hydrothermal carbonization (HTC) process of *Dimocarpus longan* peel for the development of a selective and regenerable adsorbent for cationic dye removal. The hydrochar was synthesized at 190°C with varying carbonization durations (4–12 hours) and characterized using XRD and FTIR analysis. XRD analysis revealed the formation of an amorphous carbon phase, particularly at longer HTC durations, with the most optimized structure observed at 10 hours. FTIR spectra confirmed that hydrothermal carbonization preserved the primary functional groups while modifying their intensity. Adsorption experiments using a binary dye system (malachite green/MG and rhodamine B/RhB) demonstrated that the hydrochar exhibited higher selectivity for MG, achieving a removal efficiency of 81.78%, compared to 41.88% for RhB. Desorption studies indicated that ethanol and acetone were the most effective desorbing agents, with desorption efficiencies of 18.99% and 18.14%, respectively, while water and acidic conditions showed minimal dye release. Regeneration tests revealed a gradual decline in adsorption capacity over three cycles, with removal efficiencies decreasing from 73.23% to 42.17%, indicating partial loss of adsorption sites and possible structural degradation. These findings suggest that *Dimocarpus longan* peel-derived hydrochar, particularly at 10-hour HTC processing, is a promising adsorbent for selective cationic dye removal. However, further optimization of regeneration techniques is necessary to improve its reusability and long-term performance.

Keywords

Hydrothermal Carbonization, *Dimocarpus longan* Peel, Adsorption Selectivity, Regeneration, Dyes

Received: 10 December 2024, Accepted: 15 March 2025

<https://doi.org/10.26554/ijmr.20253150>

1. INTRODUCTION

Water pollution is a critical environmental concern due to its detrimental effects on human health, terrestrial organisms, and aquatic ecosystems (Amri et al., 2024; Anggraini et al., 2024). Among the various pollutants, dyes are prominent contaminants that significantly contribute to water contamination. The rapid expansion of industries in modern society has exacerbated this issue, leading to a substantial increase in environmental pollution (Abd Al-khuder and Faiq, 2025). Cationic dyes, including malachite green (MG) and rhodamine B (RhB), pose a serious threat to water quality and can lead to various health complications. These dyes are known to cause multi-system disorders in humans and exhibit carcinogenic, teratogenic, and mutagenic properties (Palapa et al., 2023; Wen et al., 2025). Treatment methods such as coagulation, membrane filtration, and advanced oxidation processes often suffer from high operational costs, energy consumption, and secondary waste generation. As a result, adsorption has emerged as a highly effective, low-cost, and environmentally friendly approach for dye removal from aqueous

solutions. The development of sustainable, high-performance adsorbents remains a critical area of research (Duan et al., 2022; Jung et al., 2024; Lesbani et al., 2025; Siregar et al., 2021).

Biomass-derived carbonaceous materials, including biochar and hydrochar, have gained significant attention due to their abundance, cost-effectiveness, and tunable surface chemistry. Hydrothermal carbonization (HTC) is a promising method for converting biomass into hydrochar under moderate temperature and pressure conditions. This process not only enhances the material's porosity and surface functionality but also facilitates the formation of an amorphous carbon structure, improving its adsorption capabilities (Khanzada et al., 2024; Lawa et al., 2024; Xu et al., 2024).

Previous research has explored the use of *Salacca zalacca* peel as a raw material for hydrochar production and used as an adsorbent in congo red removal which resulted in an adsorption capacity of 133.333 mg/g with repeated use of 3 cycles (Hasanah et al., 2022b). Research conducted by Gabriel et al. (2023) successfully used hydrochar from *Pinus caribaea* for methylene blue removal from aqueous solution with a maximum capacity of

132.1 mg/g. Research by [Haris et al. \(2022\)](#) demonstrated that hydrochar derived from olive waste exhibited high efficiency in removing methylene blue and congo red dyes from water. Similarly, a study by [Zulfajri et al. \(2021\)](#) reported that hydrochar produced from straw mushrooms effectively removed crystal violet and methylene blue dyes, achieving removal efficiencies exceeding 90%. Although numerous studies have explored the adsorption of individual dyes, research on the selectivity of adsorbents in mixed dye systems remains limited. This aspect is crucial, as real industrial wastewater commonly contains complex mixtures of dyes rather than single-component pollutants. Evaluating adsorption selectivity in dye mixtures provides a more realistic assessment of an adsorbent's potential for practical wastewater treatment applications. MG and RhB were chosen due to their widespread industrial use and frequent occurrence in wastewater. Their distinct molecular structures and charge distributions influence adsorption interactions, making them ideal for evaluating selectivity. Studying their adsorption behavior provides insights into the adsorbent's effectiveness in treating real wastewater containing mixed dyes.

In this study, *Dimocarpus longan* peel, an agricultural waste material, was utilized as a precursor for hydrochar production via HTC with variation of process time. The adsorption efficiency, selectivity, and regeneration potential of the hydrochar were systematically evaluated. The structural and functional properties of the synthesized hydrochar were characterized using XRD and FTIR analysis to determine its suitability as an adsorbent for cationic dyes. The adsorption selectivity between malachite green (MG) and rhodamine B (RhB) was investigated, revealing a higher affinity for MG. Additionally, desorption and regeneration studies were conducted to assess the reusability of the hydrochar over multiple cycles. The findings of this study provide valuable insights into the potential of *Dimocarpus longan* peel-derived hydrochar as a sustainable and efficient adsorbent for wastewater treatment applications.

2. EXPERIMENTAL SECTION

2.1 Material and Chemicals

Dimocarpus longan peel utilized in this study was obtained from Palembang, South Sumatra, Indonesia. The chemicals used included sodium hydroxide, sodium chloride, and ethanol from EMSURE®, distilled water from Brataco, and hydrochloric acid from MallinckrodtAR®. Cationic dye powders (MG and RhB) were sourced from a textile factory. The materials were characterized using an X-Ray Rigaku Miniflex-600 Diffractometer, a Shimadzu Prestige-21 FTIR Spectrophotometer, and a Biobase Spectrophotometer UV-Visible BKUV1800PC for absorbance measurements.

2.2 Hydrothermal Carbonization Process on *Dimocarpus longan* Peel

Hydrothermal carbonization process on *Dimocarpus longan* peel adapted from [Palapa et al. \(2023\)](#). A combination of 2.5 g of *longan* peel powder and 50 mL of water was placed in a stainless-steel hydrothermal autoclave and heated in an oven at 190°C for different durations (4, 6, 8, 10, and 12 hours). Once cooled to

room temperature, the resulting solid was washed with distilled water and dried in an oven at 105°C for 24 hours to obtain the hydrochar product.

2.3 Selectivity Adsorption and Regeneration

The adsorption selectivity of malachite green (MG) and rhodamine B (RhB) dye mixtures was evaluated by introducing 0.02 g of *Dimocarpus longan* peel hydrochar into separate Erlenmeyer flasks containing 20 mL of dye solution, each with an initial concentration of 10 mg/L. The mixtures were stirred for varying adsorption durations of 15, 30, 60, 90, and 120 minutes. Following adsorption, the mixtures were filtered, and the absorbance of the filtrates was measured within the 500–700 nm wavelength range using a UV-Vis spectrophotometer. For the regeneration study, 0.02 g of *Dimocarpus longan* peel hydrochar, previously desorbed using different reagents (0.1 M hydrochloric acid, 0.1 M sodium hydroxide, ethanol, acetone, boiled water, and distilled water), was added to 20 mL of MG and RhB dye solutions with a concentration of 60 mg/L. The mixtures were stirred with a magnetic stirrer for 2 hours and subsequently separated by centrifugation. The absorbance of the filtrates was then analyzed using a UV-Vis spectrophotometer. The regenerated adsorbent was reused in the second and third cycles of desorption and adsorption to assess its reusability.

3. RESULTS AND DISCUSSION

The XRD pattern of hydrochar of *Dimocarpus longan* peel at various processing times is shown in Figure 1. Based on the data presented in Figure 1, the XRD patterns of *Dimocarpus longan* peel and its hydrochar at various hydrothermal carbonization times at 190°C exhibit diffraction peaks around 15°. Additionally, a distinct peak appears at 23.05°. The XRD analysis of hydrochar produced at 190°C reveals diffraction peaks at different hydrothermal carbonization durations: 22.58° at 4 hours, 22.22° at 6 hours, 23.1° at 8 hours, 22.27° at 10 hours, and 22.87° at 12 hours.

According to [Juleanti et al. \(2024\)](#), the broad diffraction peaks observed in the range of 15–30° indicate the formation of carbon with an amorphous phase. The XRD patterns of hydrochar obtained after 8 and 12 hours of hydrothermal carbonization show the absence of a diffraction peak around 24°, suggesting an increase in the amorphous carbon structure, as evidenced by the broadening peak at approximately 22°. Among the examined hydrochars, the optimal XRD pattern was observed for the sample subjected to hydrothermal carbonization for 10 hours. This is attributed to the presence of a diffraction peak at 22.27° with increased intensity, whereas the samples carbonized for 8 and 12 hours did not exhibit a peak around 22°, further confirming the enhancement of the amorphous carbon structure.

The FT-IR Spectra of *Dimocarpus longan* peel hydrochar at various processing times is shown in Figure 2. Based on the FTIR spectrum presented in Figure 2, a broad absorption band observed in the wavenumber range of 3500–3200 cm⁻¹ indicates the presence of hydroxyl (–OH) groups. The sharp absorption bands in the 3000–2850 cm⁻¹ region correspond to aliphatic –CH

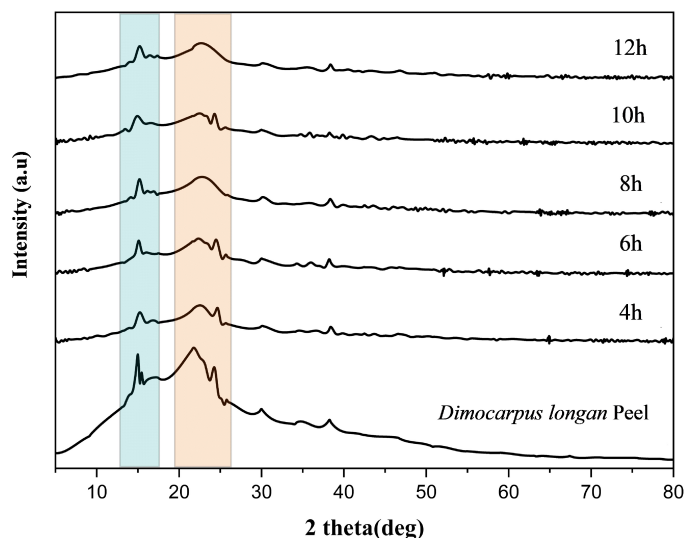


Figure 1. XRD Pattern of *Dimocarpus longan* peel hydrochar at Various Processes Time

stretching vibrations from alkane functional groups, with increasing intensity as the hydrothermal carbonization time progresses. The absorption bands in the $1760\text{--}1665\text{ cm}^{-1}$ region in the FTIR spectrum of *Dimocarpus longan* peel suggest the presence of carbonyl (C=O) groups, which exhibit enhanced intensity following hydrothermal carbonization. The peak at 1627.92 cm^{-1} corresponds to isolated C=C stretching vibrations, whose intensity decreases with longer hydrothermal carbonization durations. Additionally, absorption bands in the $1320\text{--}1000\text{ cm}^{-1}$ region indicate the presence of C-O-C bonds (Hien Tran et al., 2022; Xu et al., 2025). These FTIR findings suggest that hydrothermal carbonization at different temperatures does not alter the functional groups of the material but primarily affects their intensity, reflecting structural modifications induced by the process.

The adsorption selectivity of *Dimocarpus longan* peel hydrochar at time 10h for MG and RhB was evaluated based on the absorbance spectra of the dye mixture at different adsorption times (0, 15, 30, 60, 90, and 120 minutes), as shown in Figure 3. The initial absorbance peaks of RhB and MG appear at approximately 554 nm and 617 nm, respectively. A gradual decrease in absorbance intensity is observed over time for both dyes, indicating progressive adsorption. However, the reduction in MG absorbance is significantly greater than that of RhB, suggesting a higher affinity of the hydrochar adsorbent for MG. After 120 minutes, the removal efficiency of MG reaches 81.78%, whereas RhB is removed by only 41.88%, confirming the superior selectivity of the adsorbent towards MG. This difference in adsorption behavior may be attributed to variations in the molecular structure, charge distribution, and interaction mechanisms between the dyes and the adsorbent surface. The findings highlight the potential of *Dimocarpus longan* peel hydrochar as an effective and selective adsorbent for cationic dye removal, particularly

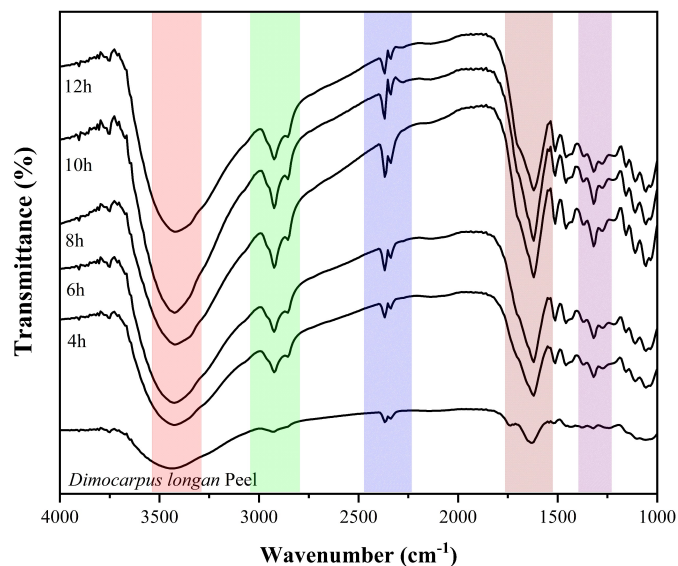


Figure 2. FT-IR Spectra of *Dimocarpus longan* peel hydrochar at Various Processes Time

for malachite green.

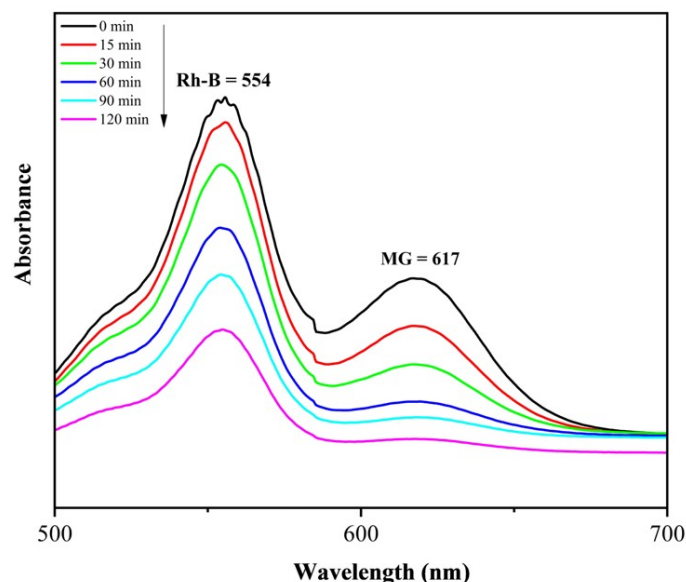


Figure 3. Selectivity Test of MG and RhB Dyes Mixture using Hydrochar 10h

The desorption and regeneration of hydrochar 10h on MG Dyes are shown in Figure 4. Figure 4(a) illustrates the desorption efficiency of hydrochar using different desorbing agents, while Figure 4(b) presents the regeneration performance of the hydrochar over multiple adsorption-desorption cycles. In the desorption study, ethanol and acetone exhibited the highest desorption efficiencies, with values of 18.99% and 18.14%, respectively. In contrast, water (2.04%) and hot water (2.79%) showed

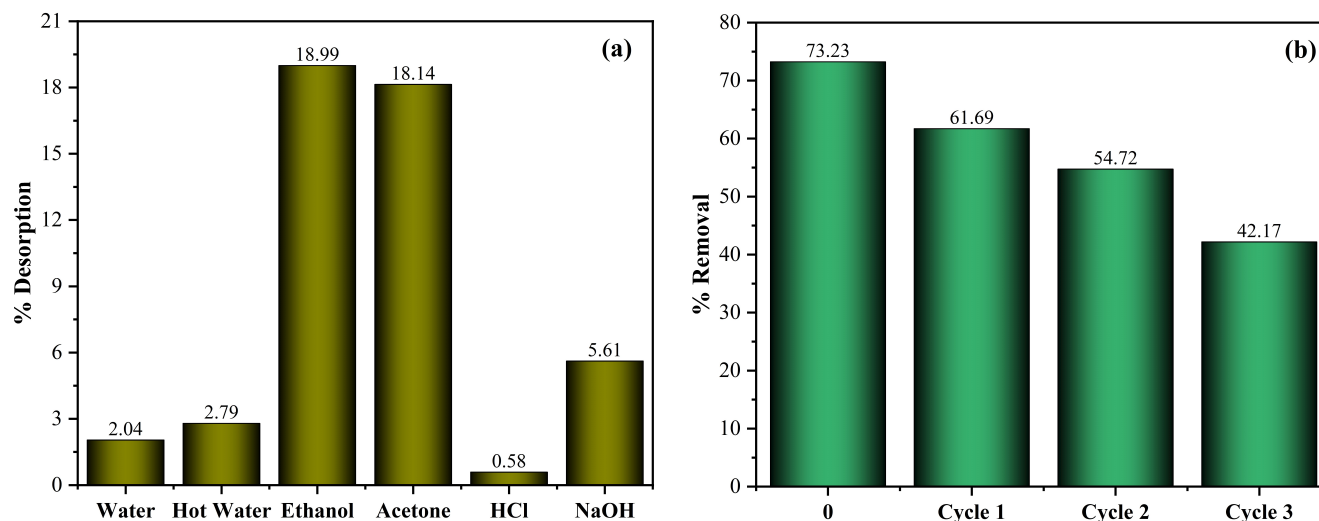


Figure 4. Desorption and Regeneration of Hydrochar 10h on MG Dyes

minimal desorption, indicating weak dye release under aqueous conditions. Hydrochloric acid (HCl) resulted in the lowest desorption efficiency (0.58%), suggesting poor interaction between the acidic medium and the adsorbed dye molecules. Sodium hydroxide (NaOH) exhibited moderate desorption efficiency at 5.61%, likely due to its ability to alter electrostatic interactions between the dye and the adsorbent surface (Hasanah et al., 2022a). The high desorption performance of ethanol and acetone suggests that organic solvents may effectively disrupt interactions between the dye molecules and the hydrochar surface, facilitating desorption.

The regeneration study (Figure 4b) demonstrates a gradual decline in adsorption efficiency across multiple cycles. Initially, the hydrochar exhibited a dye removal efficiency of 73.23%. After the first regeneration cycle, the adsorption efficiency decreased to 61.69%, followed by 54.72% in the second cycle and 42.17% in the third cycle. These findings indicate that while hydrochar from *Dimocarpus longan* peel demonstrates good adsorption performance, its reusability declines over successive cycles. The choice of desorbing agent plays a crucial role in the regeneration process, with ethanol and acetone being the most effective for dye desorption. Further optimization of regeneration strategies may enhance the longevity and efficiency of the hydrochar as a sustainable adsorbent for dye removal applications.

4. CONCLUSIONS

This study demonstrates that hydrochar derived from *Dimocarpus longan* peel via 10-hour hydrothermal carbonization is an effective and selective adsorbent for cationic dye removal, with a higher affinity for malachite green (81.78%) than rhodamine B (41.88%). XRD and FTIR analyses confirmed the formation of an amorphous carbon structure while preserving functional

groups. Desorption studies identified ethanol and acetone as the most effective regenerants, though adsorption efficiency declined from 73.23% to 42.17% over three cycles. While the hydrochar shows strong potential for dye removal, further optimization of regeneration techniques is necessary to enhance its long-term usability.

5. ACKNOWLEDGEMENT

The authors sincerely appreciate the Research Centre of Inorganic Materials and Coordination Complexes, Universitas Sriwijaya, for their valuable support, insightful discussions, and assistance with instrumental analysis throughout this study.

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