Indonesian Journal of Material Research
e-ISSN: 2987-1654
Vol. 2, No. 3, November 2024

Indonesian Journal of MATERIAL RESEARCH

Research Paper

Preparation of Hydrochar from Longan Peel: A Promising Adsorbent for Cationic Dye Removal in Aqueous Solutions

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Abstract
This study successfully prepared longan peel hydrochar via hydrothermal carbonization, characterized and evaluated for its adsorption capacity of cationic dyes malachite green (MG) and rhodamine B (RhB) in aqueous solution. Material characterization through XRD analysis revealed diffraction peaks indicative of carbonaceous content, supported by FTIR results showing −OH, C=O, and C−O−C functional groups. The BET analysis showed a surface area of 18.712 m^2/g, and SEM images displayed a heterogeneous, irregular surface morphology with spherical particles. The longan peel hydrochar exhibited adsorption capacities of 117.647 mg/g for MG and 50.505 mg/g for RhB, with MG showing greater selectivity, as demonstrated through dye mixture adsorption tests. The adsorption process was spontaneous, endothermic, and conformed to the pseudo-second-order kinetic model. Adsorption of MG followed the Freundlich isotherm, while RhB adsorption conformed to the Langmuir isotherm. Additionally, the hydrochar exhibited reusability for up to two adsorption cycles, indicating its potential as an effective adsorbent for cationic dye removal from aqueous solutions.

Keywords
Hydrochar, Longan Peel, Cationic Dyes, Adsorption

Received: 8 August 2024, Accepted: 11 November 2024
https://doi.org/10.26554/ijmr.20242344

1. INTRODUCTION

Converting low-value biomass into high-value materials remains the main goal of biomass waste valorization and bioeconomy (Fozer et al., 2024). Hydrothermal carbonization is an environmentally friendly process, simple, and efficient that converts biomass into hydrochar which is carbon-rich and rich in functional groups and porous structures. In the hydrothermal carbonization process, the biomass is placed in a closed container with water as the reaction medium and the temperature used ranges from 180 to 250°C (Fozer et al., 2024; Xu et al., 2024; Zhang et al., 2024). This process converts organic matter into hydrochar products through reactions such as hydrolysis, condensation, and aromatization (Wu et al., 2024).

Hydrochar has been utilized in various applications and shows potential as a functional ingredient such as catalytic material (Guo et al., 2024; Paula Soares Dias et al., 2024), artificial soil (Lawa et al., 2024), soil remediation (Li et al., 2021), and adsorbent (González-Fernández et al., 2024; Hasanah et al., 2022; Wibiyan et al., 2023). The use of hydrochar as an adsorbent in the adsorption process has been widely developed. The adsorption process is one of the most effective methods for adsorbing various toxic species from water and wastewater. The advantages of using adsorption methods include their simplicity in design, high performance, cost-effectiveness, reusability, and environmental friendliness (Khanzada et al., 2024). Several studies have investigated the application of hydrochar as adsorbents. Singh and Garg (2024) using hydrocarbon-derived sewage sludge as adsorbent to remove methylene blue dyes resulted in an adsorption capacity of ~190 mg/g. Hien Tran et al. (2022) prepared hydrochar from corncobs and applied it as an adsorbent for the removal of methylene blue dyes and produced an adsorption capacity of 489.560 mg/g. Hydrochar from *Lansium domesticum* and *Salacca zalacca* peel has been successfully prepared by Hasanah et al. (2022) which resulted in an increase in surface area from the raw material, effective in the process of adsorption of rhodamine B dyes, and able to be regenerated for 3 cycles.

Based on this research background, this study develops biomass waste to be used as a valuable material via hydrothermal carbonization into hydrochar. The biomass waste utilized is longan peel. The use of longan peel is motivated by its easy availability and limited utilization. Additionally, longan peel contains active compounds such as phenolic acids, polysaccharides, and flavonoids, making it a promising bioadsorbent. These compounds possess various functional groups that can directly interact with the adsorbate, making longan peel an effective

choice as an adsorbent (Mohadi et al., 2022a). The physical, chemical, and adsorption properties of the material can be improved through the conversion of biomass into hydrochar for effective application. In this research, hydrochar materials are applied to dye removal. Environmental problems due to dyes are a global problem. They exert an influence on the environment through a process known as bioamplification, which blocks light penetration in water, affects photosynthesis, and modifies the biology and chemistry of aquatic ecosystem oxygen demand (Pramanik et al., 2024; Yazid et al., 2024). These dyes are highly carcinogenic, mutagenic, and teratogenic in nature that are extremely harmful to fish, mammals, and even humans if exposed to an environment contaminated with the dyes. Therefore, it is necessary to remove dyes in aqueous solutions using effective materials (Chowdhury et al., 2024).

This research prepared hydrochar from longan peel waste via hydrothermal carbonization. The material was applied as an adsorbent in the removal of MG and RhB as cationic dyes. The factors affecting the adsorption of MG and RhB on longan peel hydrochar, including pH, contact time, concentration, temperature, selectivity of dye mixture, and regeneration ability were investigated and evaluated in detail. The adsorption data of MG and RhB were analyzed based on pseudo-first-order and pseudo-second-order kinetic models, Langmuir and Freundlich isotherm models.

2. EXPERIMENTAL SECTION

2.1 Materials

The hydrochar source was made from Longan peel waste. Water was obtained from the Bratachem Indonesia. Sodium hydroxide, ethanol, and sodium hydroxide were obtained from Merck. Hydrochloric acid was manufactured by MallinckrodtAR®. MG and RhB dyes were obtained from the textile industry.

2.2 Instrumentations

The material characterization was performed using Fourier-Transform Infrared Spectroscopy (FTIR) Shimadzu Prestige-21, X-Ray Diffraction (XRD) Rigaku mini flex-6000, surface area analyzer Quantachrome Instruments, and Scanning Electron Microscopes (SEM) analyzer SU800 Series.

2.3 Procedure

2.3.1 Preparation of Hydrochar via Hydrothermal Carbonization

A mixture of 2.5 g of longan peel powder and 50 mL of water was placed in a hydrothermal stainless-steel autoclave and heated in an oven at 250°C for 10 hours. After cooling to room temperature, the resulting precipitate was washed with distilled water and dried in an oven at 105°C for 24 hours to produce the hydrochar product, which was subsequently characterized by FTIR, XRD, BET, and SEM analyses.

2.3.2 Effect of pH Variation on Adsorption

A total of 0.02 g of hydrochar was added to a 100 mL eErlenmeyer flask containing 20 mL of a 30 mg/L solution of MG and RhB dyes. The pH was varied from 2 to 11, and the mixture was stirred for 120 minutes. Following this, the mixture was separated by centrifugation, and the absorbance of the filtrate was measured at the corresponding wavelength using a UV-Vis spectrophotometer.

2.3.3 Kinetic, Isotherm, and Thermodynamic Studies

Kinetic studies were conducted to determine the adsorption kinetics model by adding 0.02 g of adsorbent to 20 mL of dye solution, with time intervals of 0, 10, 20, 30, 40, 50, 60, 70, 90, 120, 150, 180, 210, and 240 minutes. To investigate the isotherm parameters and thermodynamic behavior of adsorption, the effect of temperature and dye concentration was evaluated. A 0.02 g sample of adsorbent was added to 20 mL of dye solution at concentrations of 25, 50, 75, 100, and 125 mg/L, and stirred for 60 minutes at temperatures of 30, 40, 50, 60, and 70°C. Dye adsorption concentrations were measured using a UV-Vis spectrophotometer, with absorbance readings taken at 617 nm for MG and 554 nm for RhB.

2.3.4 Adsorption Selectivity of MG and RhB Dye Mixture

The adsorption selectivity of the MG and RhB dye mixture was investigated by adding 0.02 g of longan peel hydrochar adsorbent to separate erlenmeyer flasks containing 20 mL of dye mixture, each with a concentration of 10 mg/L. The mixture was stirred for different adsorption times of 15, 30, 60, 90, and 120 minutes. After stirring, the mixture was filtered, and the filtrate was measured for absorbance within the 500-700 nm wavelength range using a UV-Vis spectrophotometer at each adsorption time.

2.3.5 Regeneration Process

0.02 g of longan peel hydrochar that has been desorbed using ethanol is put into 20 mL of MG and RhB dye solution with a concentration of 60 mg/L, then stirred using a magnetic stirrer for 2 hours and separated by centrifuge. The filtrate obtained was then measured for absorbance using a UV-Vis spectrophotometer. The adsorbent obtained will be reused in the 2nd and 3rd desorption and adsorption processes.

3. RESULTS AND DISCUSSION

The FTIR characteristics of longan peel hydrochar were analyzed over a band range of 4000-400 cm^{-1} (Figure 1(a)). The spectra showed the similarity of vibrations at 3435 cm^{-1} which indicates the −OH from hydroxyl and carbonyl groups of alcohol. The vibration at 2923 cm^{-1} is attributed to the vibration of C−H stretching thereby indicating the presence of methyl groups. The vibration at 1635 cm^{-1} indicates the C=O group of the amide vibrations (Sabarish et al., 2021). Figure 2(b) shows the XRD pattern of longan peel hydrochar which is at the diffraction angle of 15° and 22°. The sharp peak around 15° indicates that the material is in the crystalline phase and the broadened peak at 22° is the specific region for cellulose compounds (Palapa et al., 2023a). This shows that the hydrothermal carbonization process does not change the cellulose structure and affects the intensity

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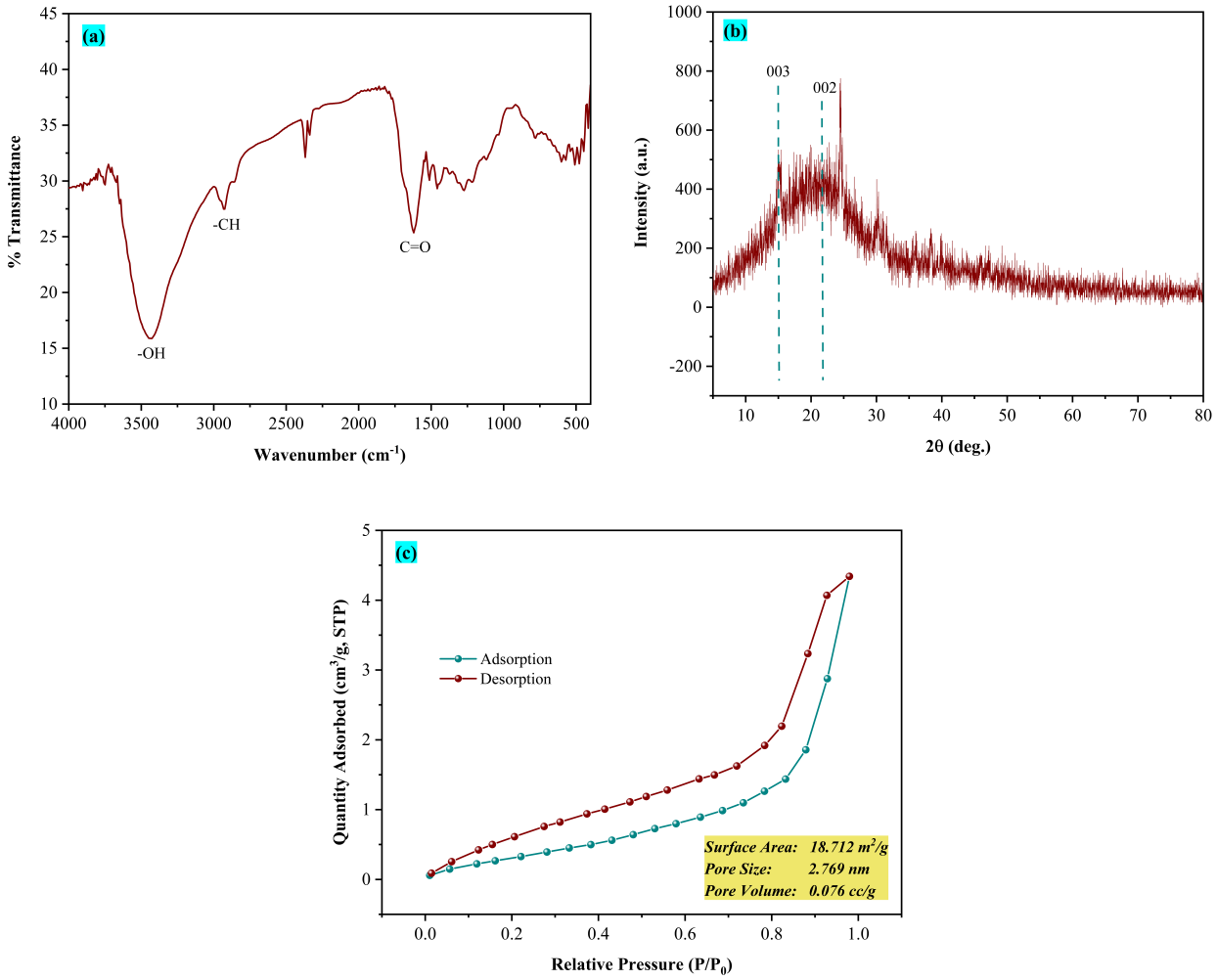

Figure 1. FTIR Spectra (a), XRD Pattern (b), and N_2 Adsorption-desorption Isotherm & BET Surface Area Analysis (c) of Longan Peel Hydrochar

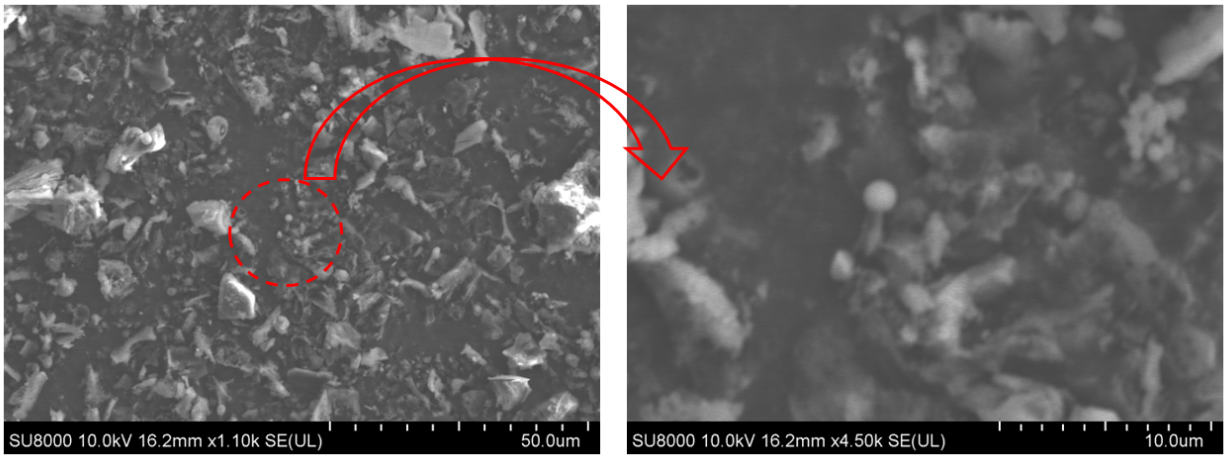

Figure 2. SEM Images of Longan Peel Hydrochar

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Table 1. Adsorption Kinetic Models

Dyes	Initial Concentration (mg/L)	Qe_{Exp} (mg/g)	Pseudo First Order (PFO)			Pseudo Second Order (PSO)		
			Qe_{Calc} (mg/g)	R^2	k_1	Qe_{Calc} (mg/g)	R^2	k_2
MG	59.430	32.504	29.635	0.993	0.022	36.765	0.995	0.001
RhB	60.226	19.658	19.792	0.990	0.024	23.095	0.993	0.001

Table 2. Adsorption Isotherm Models

Dyes	T (K)	Langmuir			Freundlich		
		Q_{max} (mg/g)	kL (mg/L)	R^2	n	kF	R^2
MG	303	117.647	0.017	0.634	1.208	2.510	0.833
	313	114.943	0.022	0.677	1.240	3.138	0.842
	323	107.527	0.031	0.759	1.301	4.151	0.849
	333	105.263	0.039	0.779	1.344	5.168	0.823
	343	109.890	0.042	0.708	1.336	5.648	0.814
RhB	303	41.841	0.040	0.961	1.720	3.046	0.912
	313	45.662	0.040	0.943	1.662	3.127	0.903
	323	47.393	0.043	0.943	1.679	3.482	0.899
	333	48.544	0.047	0.932	1.695	3.821	0.868
	343	50.505	0.050	0.916	1.704	4.158	0.901

Table 3. Thermodynamic Adsorption (C_0: 125 mg/L)

Dyes	T (K)	Qe (mg/g)	ΔH (kJ/mol)	ΔS (J/mol.K)	ΔG (kJ/mol)
MG	303	79.816	13.381	0.049	-1.374
	313	84.066			-1.861
	323	88.583			-2.348
	333	92.565			-2.835
	343	95.819			-3.322
RhB	303	71.205	9.307	0.033	-0.643
	313	75.164			-0.971
	323	78.628			-1.299
	333	81.327			-1.628
	343	84.206			-1.956

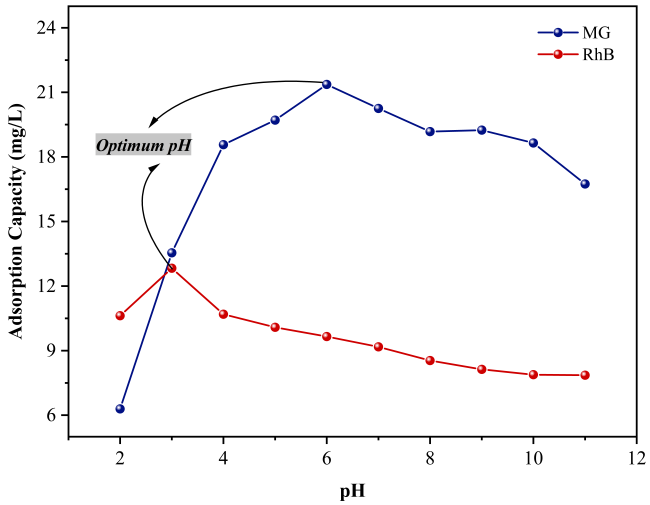

Figure 3. Effect of pH Variation on the Adsorption Process

of the diffractogram peaks which tend to have better crystallinity (Mohadi et al., 2022b).

BET surface area analysis was carried out by the N_2 adsorption-desorption process which produced the isotherm pattern in Figure 1(c). Figure 1(c) illustrates the nitrogen adsorption-desorption isotherm of longan peel hydrochar, which exhibits a type IV isotherm with a characteristic hysteresis loop, indicative of mesoporous structure (Lesbani et al., 2024). The BET analysis reveals a specific surface area of 18.712 m^2/g, a pore size of 2.769 nm, and a pore volume of 0.076 cc/g, all of which are consistent with a mesoporous structure.

Scanning electron microscopy reveals the surface morphology of longan peel hydrochar (Figure 2). The SEM images reveal the surface morphology of longan peel hydrochar at two different magnifications (1,000× and 4,500×). At lower magnification (left), the hydrochar surface appears heterogeneous with irregular shapes and a rough texture, suggesting a complex structure with varying particle sizes. This irregular morphology is typical for carbon-based adsorbents and may contribute to enhanced surface interactions with adsorbates. At higher magnification (right), the hydrochar surface shows more detailed structural features, including the presence of spherical particles and rough, uneven textures (Lin et al., 2023). These spherical particles could enhance adsorption by providing additional active sites. The heterogeneous and irregular surface morphology likely improves the hydrochar's accessibility and adsorption capacity for tar-

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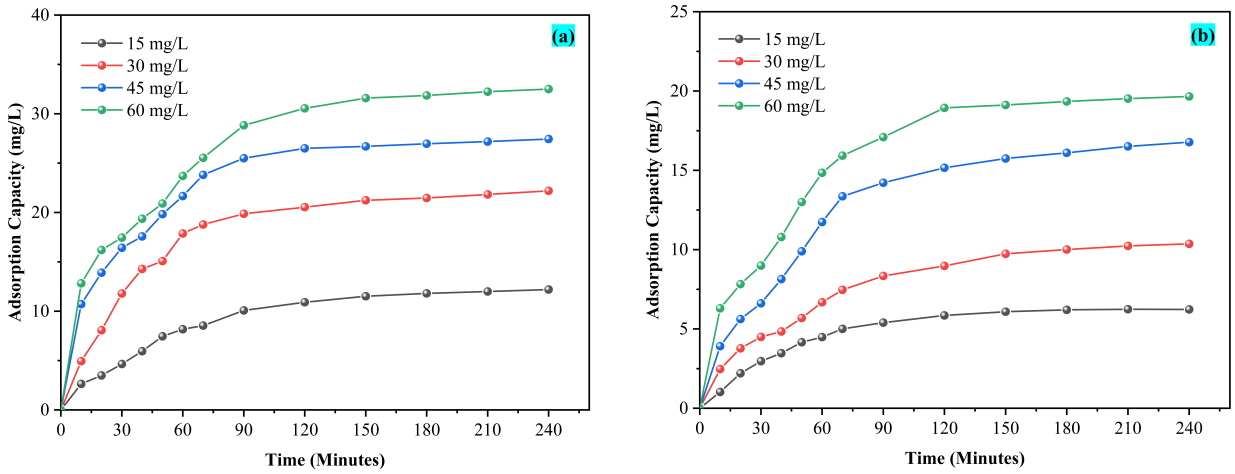

Figure 4. Effect of Contact Time on the MG (a) and RhB (b) Adsorption Process

geted pollutants, aligning well with the material's demonstrated effectiveness in dye adsorption studies.

The prepared longan peel hydrochar will be used as an adsorbent in the MG and RhB dye adsorption process. The adsorption parameters tested include the effect of pH variation, time, concentration, temperature, adsorption selectivity, and adsorbent regeneration. Analysis of adsorption data includes kinetics (pseudo-first and pseudo-second orders), isotherms (Langmuir and Freundlich), and thermodynamics. It aims to determine the ability of longan peel hydrochar to remove MG and RhB dye pollutants in an aqueous solution.

The effect of pH variation on the adsorption of MG and RhB is shown in Figure 3. Based on these data, it can be seen that the adsorption process of MG and RhB occurs optimally at pH 6 and 3, respectively, as seen from the highest adsorption capacity compared to other pHs. At the neutral pH of MG (pH 6), the negative charge of OH^- ions on the surface of the adsorbent increases, leading to electrostatic attraction between the adsorbent and malachite green dye. This results in an optimized adsorption process. In contrast, RhB dye forms zwitterions in water at pH levels above 3, causing aggregation and the formation of larger molecules. The negatively charged surface of the adsorbent leads to repulsion between the adsorbent and the dye, which results in a less efficient adsorption process (Palapa et al., 2023b).

Figure 4 shows the effect of contact time on the adsorption of MG and RhB using longan peel hydrochar. Based on the data in Figure 4, it can be seen that the concentration of dye adsorbed increases with increasing time and concentration, at more than 120 minutes the adsorbed concentration tends to be constant. So that the optimum time in the MG and RhB adsorption process occurs at 120 minutes.

The kinetic model for the adsorption process of MG and RhB including pseudo first order and pseudo second order is shown in Table 1. The kinetic model that is more likely to affect the adsorption process can be determined by looking at the coefficient of determination (R^2) value which is close to 1. Based

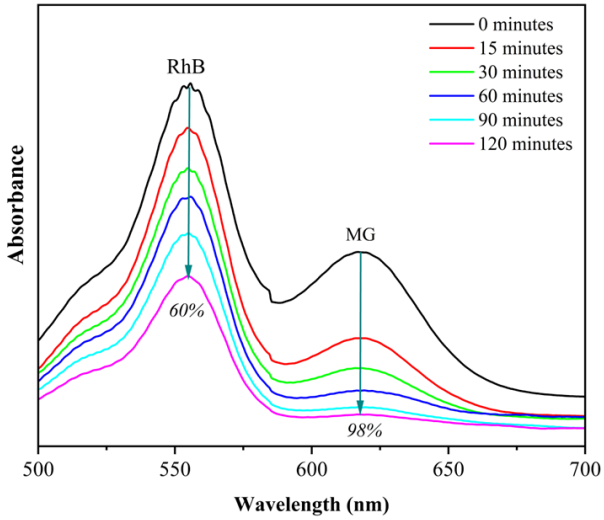

Figure 5. Wavelength Scan on Adsorption of MG and RhB Dye Mixtures

on the data in Table 2, it shows that the adsorption process of MG and RhB on longan fruit peel hydrochar is more inclined to the pseudo-second-order kinetic model, indicating that the adsorption process occurs by chemisorption and suggests that the adsorption process is most likely governed by the availability of active sites on the adsorbent and the interactions between the dye molecules and the adsorbent surface (Iqbal et al., 2025; Normah et al., 2024).

The adsorption isotherm models determined were Langmuir and Freundlich's isotherm is shown in Table 3. The MG adsorption process tends to follow the Freundlich isotherm model, while RhB follows the Langmuir isotherm model as seen from the linear regression value which is close to 1. The Langmuir model assumes monolayer adsorption on a homogeneous surface with no interaction between adsorbed molecules, suggesting a

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fixed number of identical sites. Meanwhile, the Freundlich model is an empirical equation that accommodates heterogeneous surface energies, allowing for multilayer adsorption and varying adsorption intensities on different sites (Ahmad et al., 2023). The adsorption capacity obtained in the adsorption process of MG and RhB using longan peel hydrochar was 117.647 mg/g and 50.505 mg/g, respectively. This shows that longan peel hydrochar material can be used as an effective material in removing MG and RhB dyes in aqueous solutions.

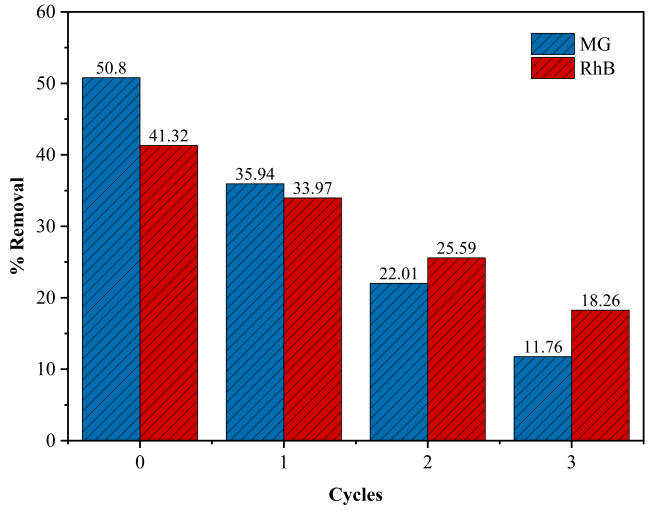

Figure 6. Regeneration of Longan Peel Hydrochar on MG and RhB Adsorption

The thermodynamic data of adsorption is shown in Table 2. The positive value of entropy energy (ΔS) indicates that there is increased randomness in the adsorption process. The enthalpy values (ΔH) in the range between 5-40 kJ/mol with positive enthalpy values and negative Gibbs free energy (ΔG) indicate that the adsorption process is endothermic, spontaneous, and indicates physical adsorption (Anggraini et al., 2024).

Figure 5 displays the wavelength scan on the adsorption of the MG and RhB dye mixture. This aims to determine which dye is more selective in terms of percent removal. Based on the wavelength scan, it can be seen that the percentage of MG removal is greater even reaching 98%, this shows that MG is more selective than RhB using longan peel hydrochar. The higher selectivity of MG over RhB in adsorption using longan peel hydrochar may be due to differences in the molecular structure, size, or charge of the dyes, which influence their interaction with the hydrochar surface. MG as a cationic dye, likely forms stronger electrostatic interactions with functional groups on the hydrochar, particularly if the surface contains negatively charged or polar sites that enhance its affinity for positively charged molecules. This interaction could result in higher adsorption efficiency and selectivity for MG compared to RhB.

Regeneration of longan peel hydrochar in the MG and RhB adsorption process obtained data for each cycle in Figure 6. It can be seen that there is a decrease in the percent removal of each regeneration cycle. The decrease in removal efficiency may be due to the reduction of active sites and available pores on the material caused by desorption using reagents during the regeneration process (Yazid et al., 2024). In the third regeneration cycle, the results show that the percent removal is below 50% from cycle zero, so it is found that longan peel hydrochar can be regenerated in the MG and RhB adsorption process for 2 cycles with a percent removal of 22.01% and 25.59% respectively.

4. CONCLUSIONS

Longan peel hydrochar was successfully prepared via hydrothermal carbonization, yielding adsorption capacities of 117.647 mg/g for malachite green (MG) and 50.505 mg/g for rhodamine B (RhB). The selectivity test confirmed that MG is more selectively adsorbed than RhB in a dye mixture. The adsorption process was spontaneous, endothermic, and aligned with the pseudo-second-order kinetic model. Adsorption of MG followed the Freundlich isotherm, while RhB adsorption conformed to the Langmuir isotherm. Additionally, the longan peel hydrochar demonstrated reusability for up to two regeneration cycles, indicating that is a promising adsorbent for the effective removal of cationic dyes from aqueous solutions.

5. ACKNOWLEDGEMENT

The authors express their gratitude to the Research Centre of Inorganic Materials and Coordination Complexes at Universitas Sriwijaya for their valuable contributions through discussions, support, and instrumental analysis provided during this research.

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