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Research Paper

Preparation and Application of Biochar from *Areca catechu* L. Peel for Malachite Green and Reactive Blue Dyes Removal

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Abstract
The utilization of *Areca catechu* L. fruit peel waste as biochar offers a sustainable solution for waste management while enhancing the material's value as an efficient adsorbent. Biochar from *Areca catechu* L. fruit peels was processed through pyrolysis for 2 hours at temperature of 500°C. This study investigates the use of biochar from *Areca catechu* L. peel as an adsorbent for Malachite Green (MG) and Reactive Blue (RB) dyes in a batch system, with a focus on analysing the kinetic adsorption processes. The biochar was characterized using XRD, FTIR, SEM and BET. BET analysis was conducted, which shows that the surface area of biochar from *Areca catheca* L. fruit peel after pyrolysis treatment was 29.52 m^2/g. The adsorption of MG by the biochar from *Areca catechu* L. peels was found to be greater than that of RB. The biochar showed an adsorption capacity of 16.113 mg/g for MG, while its capacity for RB was 15.499 mg/g. The adsorption process of biochar from *Areca catechu* L. peels for both MG and RB dyes follows a pseudo-second-order kinetic model.

Keywords
Biochar, *Areca catechu* L., Adsorption, Malachite Green, Reactive Blue

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1. INTRODUCTION

The textile sector is a significant contributor to environmental (Ali et al., 2024). The textile industry releases a substantial amount of dye waste into the environment (Salih et al., 2022). The rapid advancement of industries involving dyes, if not accompanied by proper waste treatment, can pose risks to water quality and ecosystems (Kosale et al., 2024). Examples of dyes utilized in the textile industry include Malachite Green (MG) and Reactive Blue (RB). These dyes are classified as cationic and anionic, and possess genotoxic, teratogenic, and carcinogenic properties. Their potential for causing harm to vital organs such as the liver, kidneys, heart, lungs, and skin is of particular concern if they enter the human body (Eltaweil et al., 2020; Bouzikri et al., 2020). A prevalent method employed for dye treatment is adsorption, a process that is characterized by its efficiency, cost-effectiveness, and user-friendly operation (Wang et al., 2022). The mechanism of adsorption involves the attraction of pollutants to the surface of adsorbent materials, facilitating the elimination of harmful substances from waste water (Alver et al., 2020).

Biochar is a type of adsorbent that is finds application in the adsorption process. Biochar is a carbon-dense material generated through the thermal decomposition of biological waste under limited oxygen conditions. This material has multiple applications, including in microbial development, soil enhancement, and the removal of dye pollution (Aziz et al., 2023).

Research on biochar production has been conducted by utilising various biomass sources, such as rice husk (Ahmad et al., 2020), rambutan peel (Islam et al., 2019), duku peel (Awogbemi and Kallon, 2023), durian peel (Chua et al., 2023), and corn klobot (Wijitkosum, 2022). One other biomass source that has the potential to be used in biochar production is areca nut *Areca catechu* L. husk. Areca nut shells contain lignocellulose which makes up about 65-80% of the total weight of the fruit (Vikraman et al., 2022). However, the utilisation of areca nut shells is still limited, as they are generally only considered as waste. This agricultural waste has a slow decomposition rate and is less effectively used as fertiliser. Therefore, the conversion of areca nut shells into biochar can be a sustainable solution to reduce the impact of the resulting environmental waste (Manjunath et al., 2024). The high lignocellulose content of areca nut

shells can be optimised in the biochar production process, thereby increasing its environmental and economic benefits.

Recent research has examined the modification of areca nut (*Areca catechu* L.) shells into carbon-based materials such as biochar and hydrochar, which have potentially used in the adsorption process of synthetic dyes from wastewater (Adawiyah et al., 2024). Biochar was obtained through pyrolysis process, while hydrochar was synthesised using hydrothermal carbonisation method. XRD analysis validated the presence of carbon in both materials with an amorphous structure. Characterisation results using FTIR showed the existence of major functional groups, such as ester, hydroxyl and carboxyl, that have an important role in the adsorption process. Specific surface area measurements using the BET (Brunauer-Emmett-Teller) method showed values of 82.584 m^2/g for biochar and 77.618 m^2/g for hydrochar. Adsorption tests showed that hydrochar had an adsorption capacity of 40.515 mg/g for Congo Red dye, while biochar showed a capacity of 40.616 mg/g, which was much higher than that of unmodified areca nut shell at 23.168 mg/g. Moreover, both materials were able to maintain their structure and performance after three regeneration cycles, indicating their potential as sustainable adsorbents to address industrial dye pollution.

The objective of this study is to synthesise biochar from Areca catechu fruit peel using a concise and effective method and to examine its performance in adsorbing Malachite Green and Reactive Blue dyes. To understand the characteristics of the biochar produced, in-depth analyses of the crystalline phase, functional groups, and morphology were conducted using FTIR, XRD, and BET analysis. The present study also investigated the core variabels that affecting the adsorption process, including contact time, with a view to understanding the mechanism of adsorption kinetics.

2. EXPERIMENTAL SECTION

2.1 Chemicals and Instrumentation

All of the chemicals used in this research are distilled water (H_2O) obtained by PT. Dira Sonita, Hydrogen Cloride (HCl), Natrium Hydroxide (NaOH), Natrium Cloride (NaCl), Malachite Green Dye ($C_{23}H_{25}N_2Cl$), Reactive Blue Dye ($C_{40}H_{23}Cl_2N_{15}Na_6O_{19}S_6$) and supplied by Merck and Areca fruit peel (*Areca catechu* L.) from Pangandaran, West Java. Equipment utilized includes XRD Rigaku Miniflex-600, FTIR Shimatdzu Prestige-21, Quantachrome NOVA 4200e Surface Area, SEM-EDX SU 8000 series, and spektrofotometer UV-Vis Orion Aquamate 8000.

2.2 Preparation of Biochar from *Areca catechu* L. Fruit Peel

Areca catechu L. fruit peels were washed to clean impurities and then dried under the sun three days to minimize moisture content. They were then oven-dried for 12 hours at 800°C to remove the moisture and any volatile compounds. The hides were ground into small particles and then pyrolysed in a furnace at 500°C with a nitrogen flow of 1×10^3 m^3/hour for three hours. The resulting biochar was washed and redried in a hot air oven at 90°C for 12 hours (Adawiyah et al., 2024).

2.3 Kinetcs Adsorption

2.3.1 Adsorption of Malachite Green (MG) and Reactive Blue (RB)

Experiments were conducted to investigate the adsorption behavior of MG in a time range of 10 to 180 minutes. The initial concentration of MG was 20 mg/L, and 0.02 g of adsorbent was used in 20 mL of dye solution. Following the adsorption process, the concentration of MG and RB, UV-Vis spectrophotometry was used to analyze the filtrate as previously described.

2.3.2 Data Analysis

The adsorption mechanism influenced by the variation of contact time, is utilised to evaluate the adsorption kinetics. The latter can be calculated using pseudo first-order and pseudo second-order models as follows Equations 1 & 2 (Ali et al., 2018).

$$\ln(q_e - q_t) = \ln q_e - k_1 t \tag{1}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{2}$$

Where k_1 = pseudo-first-order rate constant (min^{-1}), qe = adsorption capacity at equilibrium (mg/g), qt = adsorption capacity at time, t = time (minutes), and k_2 = pseudo-second-order constant ($mg/g.min^{-1}$).

3. RESULT AND DISCUSSION

The crystallinity characterization of *Areca catechu* L. fruit peel biochar and *Areca catechu* L. fruit peel powder was performed using XRD. The diffractogram data, which showcases a comparison between the two materials, is presented in Figure 1. The wide peak detected in the 16-25° angle range is indicative of amorphous compound characteristics, as previously reported by Pariyar et al. (2020). The peak at the angle of 21-26° in the XRD pattern of the biochar is associated with the carbon (002) plane. A similar carbon plane at 22.4° (002) is exhibited by *Areca catechu* L. fruit peel. Other carbon planes are indicated at angles of 16.7° and 60.77°, corresponding to the (003) and (110) planes, respectively (Mohadi et al., 2021).

The FTIR spectra on Figure 2. were observed at wavenumbers ranging from 500 to 4000 cm^{-1}, demonstrating various bond types and absorption regions. A significant absorption peak was observed at 3440 cm^{-1} in *Areca catechu* L.

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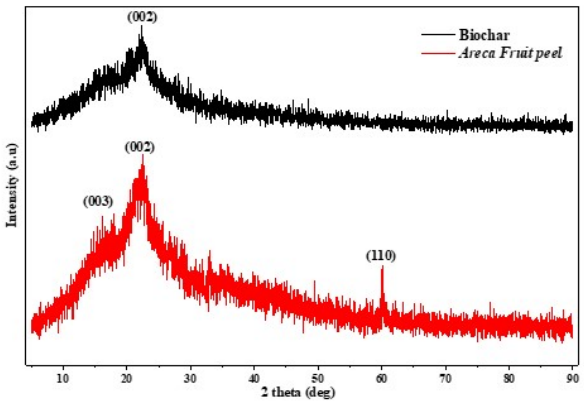

Figure 1. *Areca catechu* L. Fruit Peel and Biochar XRD Patern

fruit peel, indicative of −OH stretching and bending vibrations from water present in the material. The peak at 1610 cm^{-1} indicated aromatic C=C stretch, conjugated ketone C=O. The absorption region around 2930 cm^{-1} indicated symmetric C−H stretching from aliphatic compounds. The absorption region at 1422 cm^{-1} indicated C=C stretching, suggesting the existence of lignin and aromatic compounds. Absorption at 1144 cm^{-1} indicated ester C−O−C groups from cellulose and hemicellulose, as supported by absorption at 1068 cm^{-1} indicating symmetric C−O−C ester stretching in aliphatic groups (Janu et al., 2021)

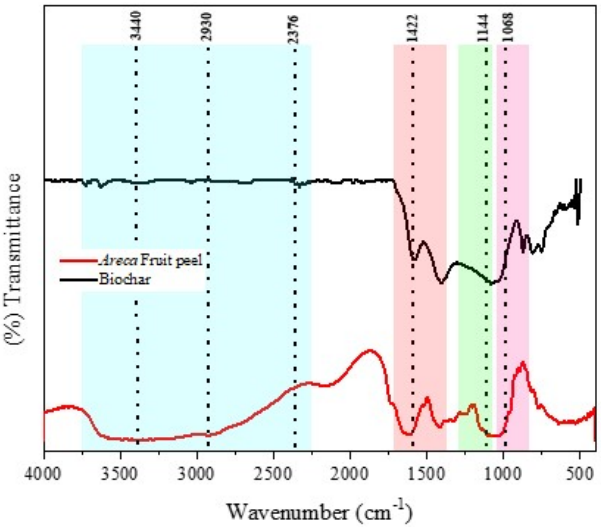

Figure 2. *Areca catechu* L. Fruit Peel and Biochar FTIR Spectrum

According to the FTIR spectra, the vast absorption inside the 3000-3600 cm^{-1} area, indicated −OH groups from water, disappeared after pyrolysis. The existence of C=C bonds were indicated from the absorption at 1615 cm^{-1} and the peak at 2931 cm^{-1} was frequently associated with −CH groups in extended aliphatic chains (Siregar et al., 2021). Furthermore, the 770-870 cm^{-1} region has been identified as a marker for aromatic −CH groups (Manjunath et al., 2024). Aromatic C=C groups were observed at around 1400 cm^{-1}, while aliphatic ester C–O–C groups were detected in the 1040 cm^{-1} absorption region (Bolbol et al., 2019). The intensity of the bands observed in Areca fruit peel biochar could be attributed to an increase in functional groups percentage with a concomitant loss of other groups mass due to pyrolysis.

The surface morphology of the biochar was evaluated by using SEM analysis. Figure 3. Shows the result of the SEM analysis. The irregular and distorted pores with an uneven structure are shown in Figure 3(a), indicating unopened pores on the Areca fruit peel. In contrast, Figure 3(b) illustrates the morphology of *Areca catechu* L. fruit peel biochar with pores of varying sizes. The formation of pores of different sizes is attributed to the evaporation or release of organic materials during high-temperature pyrolysis (Gupta et al., 2018).

Figure 4. Shows the N_2 adsorption-desorption curve shows a type IV isotherm, which is characteristic of mesoporous materials with pore sizes ranging between 2-50 nm. This is confirmed by the existance of hysteresis in the desorption profile, indicating the existence of a regular pore structure. The tested biochar's surface area shown in Table 1. is 29.52 m^2/g, with 0.130 cc/mg a pore volume and an average pore size, which corresponds to 15.39 nm and in the mesoporous range. This mesoporous structure optimises the diffusion of adsorbate molecules into the pore, thereby increasing the adsorption capacity. However, the relatively low surface area of biochar compared to conventional activated carbon suggests that further activation may be required to improve adsorption efficiency.

Table 1. BET Analysis Result of Biochar

Material	Surface area (m^2/g)	Average pore size (cc/mg)	Average pore volume (nm)
Biochar	29.52	0.130	15.39

The pHpzc value of biochar and Areca fruit peel were proven by shaking 0.05 g of the material in 0.01 M NaCl solution, where the pH of the mixture was modified using 0.01 M NaOH and 0.01 M HCl for a period of three hours (Jedynak and Charmas, 2021). The pHpzc values of biochar and *Areca catechu* L. fruit peel powder are shown in Figure 5, with values of 6.84 and 6.63, respectively. This value indicates that at this pH, the material's surface is neutral. If the pH of the solution is lower than the pHpzc value, the adsorbent surface is positively charged, allowing it to attract anions. In contrast, at pH levels higher than the pHpzc, the biochar surface becomes negatively charged (Bernal et al., 2017). The pHpzc value is a crucial parameter in adsorbents,

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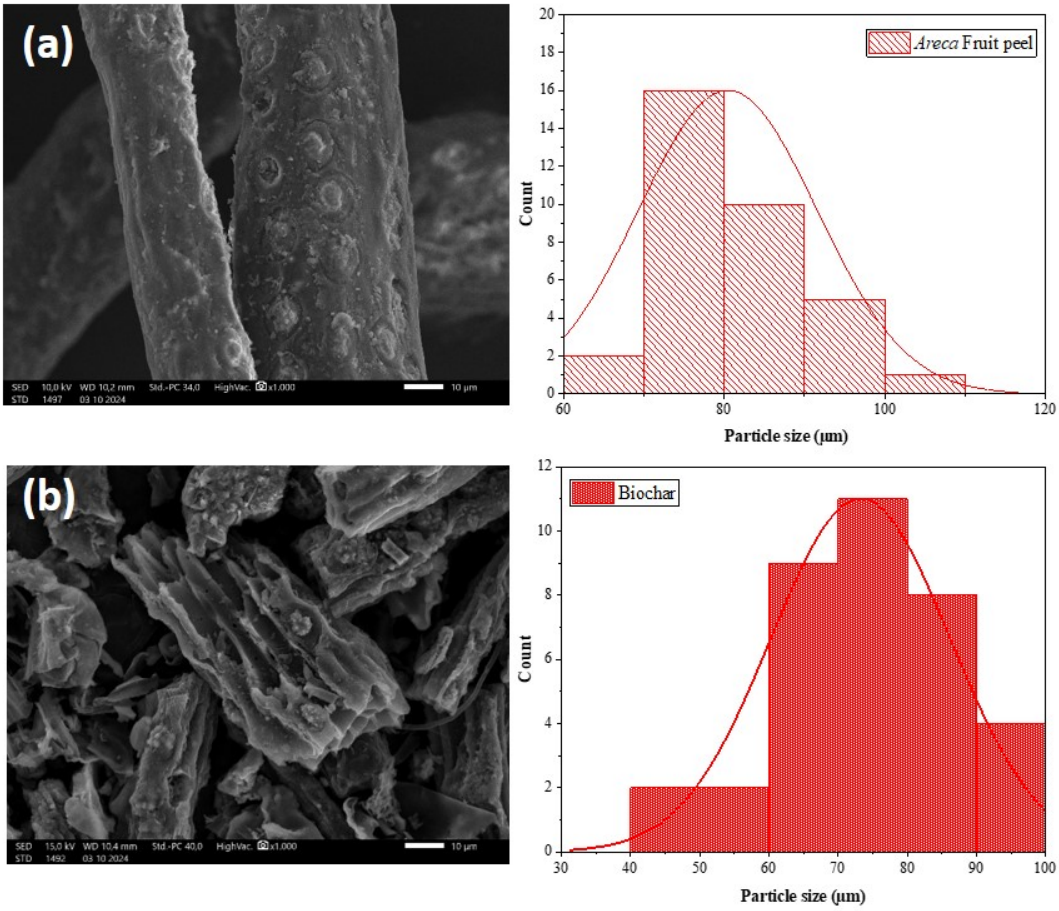

Figure 3. Morphology and Particle Size Distrubution of *Areca catechu* L. fruit peel (a) Biochar (b)

as it significantly impacts adsorption efficiency and can vary between materials (Al-Maliky et al., 2021).

The impact of contact time during adsorption is shown in Figure 6a, where the dye uptake gradually increases until equilibrium is reached. In this research, equilibrium in the adsorption of MG using *Areca catechu* L. fruit peel powder and biochar was achieved after 40 minutes, with no significant increase in adsorption capacity observed after this time. The highest adsorption capacity recorded after 180 minutes revealed that biochar exhibited a capacity of 16.113 mg/g, while Areca fruit peel powder attained 17.564 mg/g. This outcome indicates that *Areca catechu* L. fruit peel powder possesses a higher adsorption capacity than biochar, a phenomenon that can be attributed to the lower pHpzc value of *Areca catechu* L. fruit peel powder compared to biochar, resulting in its surface being more negatively charged at the natural pH of the MG solution.

Figure 6b. illustrates the role of contact time in adsorption capacity of RB into Areca fruit peel and Areca fruit peel biochar. The adsorption process was marked with a sharp initial rise, gradually transitioning to a slower adsorption

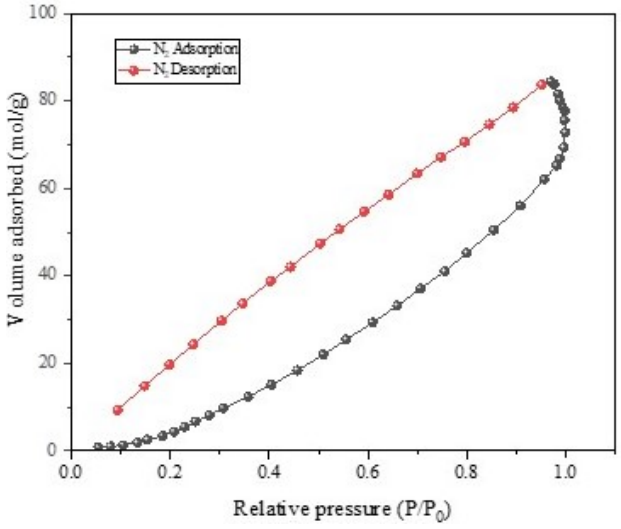

Figure 4. N_2 Adsorption and Desorption Isotherm of Biochar

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Table 2. Kinetic Parameters for MG dye

Adsorbent	Initial Concentration (mg/L)	Qe_{exp} (mg/g)	Qe_{calc} (mg/g)	PFO R^2	PFO k_1	PSO Qe_{calc} (mg/g)	PSO R^2	PSO k_2
Areca catechu L. fruit peel	20.38	17.564	6.001	0.8114	0.0313	17.921	0.9998	0.0161
Biochar	20.38	16.113	3.737	0.9205	0.0387	16.390	0.9998	0.0245

Table 3. Kinetic Parameters for RB dye

Adsorbent	Initial Concentration (mg/L)	Qe_{exp} (mg/g)	Qe_{calc} (mg/g)	PFO R^2	PFO k_1	PSO Qe_{calc} (mg/g)	PSO R^2	PSO k_2
Areca catechu L. fruit peel	100.67	10.00	8.290	0.982	0.013	10.384	0.982	0.002
Biochar	100.78	15.499	14.057	0.992	0.0022	18.410	0.996	0.001

rate until equilibrium was reached. This suggests that most the existing active sites on the adsorbents were taken within the first 150 minutes of the process.

At 180 minutes, the adsorption capacity of Areca fruit peel and Areca fruit peel biochar was 10.000 mg/g and 15.499 mg/g, indicate the biochar has higher adsorption efficiency. This is consistent with the pseudo-second-order (PSO) kinetic model, which was best fitted ($R^2 = 0.996$ for biochar and $R^2 = 0.982$ for *Areca catechu* L. fruit peel) in coparison to the pseudo-first-order (PFO) model. The greater *qe* calculated (18.410 mg/g in biochar and 10.384 mg/g in *Areca catechu* L. fruit peel) in the PSO model confirms that adsorption is largely controlled by chemisorption mechanisms. The adsorption kinetics were analyzed using the PFO and PSO models, calculated based on Equations 1 and 2.

Improved performance of biochar can be attributed to the improved surface properties it has, i.e., higher surface area and improved porosity upon carbonization. Additionally, the pHpzc of biochar is higher than the raw *Areca catechu* L. fruit peel, and this makes electrostatic interaction between Reactive Blue dye and experimental pH more favorable. Overall, the results indicate that *Areca catechu* L. fruit peel biochar is a more effective adsorbent compared to raw *Areca catechu* L. fruit peel for the removal of Reactive Blue dye, largely due to its higher adsorption capacity and better kinetic compliance with the pseudo-second-order model.

Adsorption process demonstrates an increase over time until equilibrium is ultimately achieved. Reactive blue dye adsorption using *Areca catechu* L. fruit peel and *Areca catechu* L. fruit peel biochar reveals distinct equilibrium conditions. The biochar attains its maximum capacity following a contact time of 180 minutes, whereas the *Areca catechu* L. fruit peel reaches equilibrium at 150 minutes. Subsequent to this, the absorption of the *Areca catechu* L. fruit peel experiences a decline.

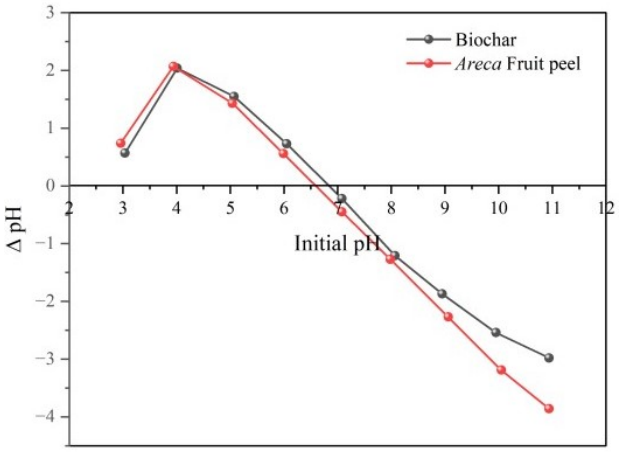

Figure 5. pHpzc of *Areca catechu* L. Fruit Peel and Biochar

Biochar demonstrated capacity adsorption of 15.499 mg/g, while the capacity of *Areca catechu* L. fruit peel powder was 10.000 mg/g. The enhancement in adsorption capacity until reaching equilibrium is ascribed to the accessibility of active sites on the surface of the adsorbent that are capable of interacting with the dye during the contact process.

The mechanism and rate of adsorption of biochar and *Areca catechu* L. fruit peel powder on Malachite Green (MG) and Reactive Blue (RB) dyes, adsorption kinetics analysis based on contact time variation was conducted. Pseudo-

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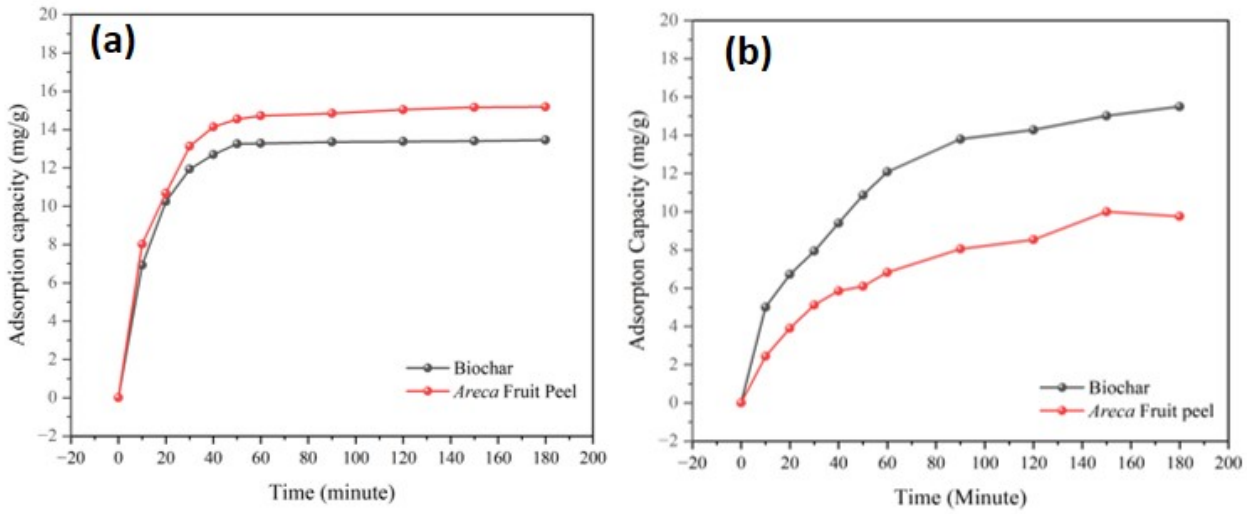

Figure 6. Kinetic Adsorption of Malachite Green (a) Reactive Blue (b) on *Areca catechu* L. Fruit Peel and Biochar

first-order and pseudo-second-order kinetic models were applied to determine the most suitable mechanism, with the results presented in Tables 2 and 3. The evaluation of the data indicated that the adsorption of MG by both adsorbents was more in line with the pseudo-second-order model.The adsorption process of MG by biochar followed the pseudo-second-order model, as indicated by the coefficient of determination (R^2) value of 0.999, which is very close to one.

Furthermore, the theoretical adsorption capacity derived from this model exhibited a strong correlation with the experimental adsorption capacity, as demonstrated in Table 2. The experimental adsorption capacity of biochar was documented at 16.390 mg/g, which closely approximates the theoretical value of 16.113 mg/g. In a similar manner, although the pseudo-first-order model exhibited a relatively high correlation for *Areca catechu* L. fruit peel powder, the pseudo-second-order model provided a superior fit. This suggests that although physical interactions may occur, the predominant mechanism involved in adsorption is chemisorption, as substantiated by the higher R^2 value (0.999). The theoretical adsorption capacity of 17.921 mg/g is closely aligned with the experimental capacity of 17.564 mg/g, further validating the model's predictions.

As demonstrated in Table 3, the adsorption kinetics of *Areca catechu* L. fruit peel biochar for RB dye align with pseudo-second-order kinetics and adsorption capacity is 15.499 mg/g approaching the theoretical value of 18.416 mg/g. This finding suggests that biochar exhibits a higher adsorption capacity according to pseudo-second-order kinetics calculations, but requires a longer contact time. A similar observation was made for *Areca catechu* L. fruit peel, where the experimental adsorption capacity of 10.000 mg/g similiar to the theoretical value of 10.384 mg/g, as determined by pseudo-second-order kinetics. The pseudo-second-order model exhibited a more robust linear correlation, as evidenced by its higher R^2 value for both *Areca catechu* L. fruit peel and BC adsorbents. This finding indicates that the predominant adsorption mechanism is surface diffusion and chemisorption (Srivatsav et al., 2020).

4. CONCLUSIONS

The characterization of *Areca catechu* L. fruit peel biochar reveals an XRD pattern of amorphous compounds rich in carbon. The analysis of functional groups through Fourier-transform infrared spectroscopy (FTIR) reveals the presence of hydroxyl, carboxyl, acid, and ester functionalities, which are predominant in the biochar. The biochar's morphology indicates non-uniform pores, and the pyrolysis process has been proven to increase the material's surface area, with a BET surface area analysis of 29.52 m^2/g. The absorption capacity of biochar in this study for MG and RB is 16.113 mg/g and 15.499 mg/g, respectively, and follows a pseudo-second-order kinetic model.

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