

Preparation of Low-Cost Activated Carbon from Spent Coffee Grounds for Rhodamine B Removal

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Abstract—A low-cost coffee-based activated carbon for Rhodamine B (RhB) removal was investigated. Spent coffee grounds obtained from a local coffee shop in Pathio, Chumphon, Thailand, were utilized as the raw material to produce biochar. The coffee grounds were pre-treated by washing with 1M H₂SO₄ before carbonization at 600 °C for 2 h under a limit-oxygen atmosphere. The acid washing process was found to be a promising method for preparing activated carbon without requiring an inert gas flow during the carbonization. This approach resulted in activated carbon free of ash after carbonization. The carbonized coffee grounds were activated using a microwave-assisted process with KOH as the activating agent. The performance of the prepared activated carbon for Rhodamine B adsorption was evaluated using the batch method. Key parameters, including initial concentration of Rhodamine B (3–11 mg/L), pH (3–11), and contact time (15–150 min), were optimized. The maximum adsorption capacity was 16 mg/g of adsorbents. The optimal pH for Rhodamine B adsorption was found to be 7. The adsorption of Rhodamine B onto the adsorbent fits a Langmuir isotherm and a pseudo-second-order kinetic model. The study revealed that the prepared activated carbon from waste coffee grounds is an efficient and affordable solution for removing Rhodamine B contaminants from the water system.

Keywords—activated carbon, Rhodamine B, KOH, limit-oxygen carbonization, microwave activation, acid washing

I. INTRODUCTION

Rhodamine B (RhB) is a synthetic dye that is widely used in various industries, such as textiles, leather, paper, cosmetics, food coloring, stamp pad inks, ball pens, fireworks, and dyeing mosquito coils. The non-biodegradable nature of Rhodamine B in wastewater can cause a tremendous effect on both humans and animals as it is classified as a neurotoxic, carcinogenic, and respiratory irritant dye [1]. It can cause infections in the eyes, skin, gastrointestinal system, and respiratory system. Moreover, Rhodamine B can also have a significant impact on the growth and development of aquatic organisms [2–4]. The Quality Standard (MAC-QS) has set the maximum concentration of Rhodamine B at 140 µg/L as the allowable limit for exposure to water to prevent harm to aquatic systems. To prevent long-term toxic effects, the Annual-Average-Quality Standard (AA-QS) has set the permissible level of Rhodamine B at 14 µg/L [1, 4, 5]. Therefore, it is vital to develop efficient methods for removing Rhodamine B from the environment to minimize its risks to humans, animals, and aquatic ecosystems.

There are several methods that have been reported for the removal of Rhodamine B from aqueous solutions, including photocatalytic degradation, membrane separation, and

oxidation methods. However, the adsorption method is prominent due to its effectiveness, cost-effectiveness, and ease of operation. Various adsorbent materials have been employed for dye removal, such as zeolite, clays, polymer composites, silica, and activated carbon [6, 7]. Among these, activated carbon is one of the most frequently used due to its remarkably high surface area and high capability to adsorb Rhodamine B [8, 9]. Activated carbon can be prepared from various sources of materials. The promising materials that are widely used are derived from biomass and industrial waste and have been reported for their potential in various applications [10, 11]. Recent literature has reported that carbonaceous materials for Rhodamine B removal can be derived from biomass and waste material [12–15]. This could be a way to utilize waste to produce valuable activated carbon for Rhodamine B removal from aqueous solution.

II. LITERATURE REVIEW

Various procedures for the preparation of activated carbon for Rhodamine B adsorption in aqueous solution have been reported. Biowastes, such as spent coffee grounds, lignocellulosic, sawdust, cotton stalks, banana peel, etc. [16–20] were one of the major precursors for preparing activated carbon because they are inexpensive and readily available for renewable carbon sources. Spent coffee ground-based activated carbon for the adsorption of Rhodamine B has been previously investigated with different processes. Hong-Ming Chen *et al.* [20] prepared activated carbon from waste coffee by drying the raw material at 80 °C for 10h and then carbonizing it under an argon atmosphere at 900 °C for 1h. The samples were then activated with KOH at 65 °C for 16h and dried after the activation process at 100 °C for 20h before being annealed at 900°C for 1h under argon flow. The resulting materials were washed with hydrochloric acid and deionized water until a pH of 7 was reached. They were then filtered and dried before use. Activated carbon obtained from this study was capable of adsorbing Rhodamine B at a maximum capacity of 83.4 mg/g of carbon at an initial dye concentration of 100 mg/L. Wirawan *et al.* [21] prepared coffee-based activated carbon by first soaking the coffee grounds in boiled water and then washing, neutralizing, filtering, and drying them at 105°C for 3h. The pretreated coffee grounds were then carbonized at 500°C in a muffle furnace for 45min under a low-oxygen condition. The obtained material was chemically activated by soaking in H₃PO₄ for 24h. The mixture was then filtered, washed with water until neutralized, and dried for 3h at 105°C. Adsorption studies of Rhodamine B were conducted using batch method,

and the maximum adsorption capacity was found to be 4 mg/g of carbon.

The preparation of activated carbon from coffee residue using microwave radiation was demonstrated by Juengsamarn *et al.* [22]. The study was conducted by first washing the materials with water until a pH 7 was reached, followed by drying at 100 °C for 12 h. Dried coffee grounds were carbonized at 400, 500, and 600°C for 1h under nitrogen gas flow. The resulting material was chemically activated with 40% v/v H₃PO₄ (1 g of carbonized coffee ground per 20 mL of H₃PO₄) for 24 h. The activated coffee residue was then physically activated with microwave radiation at 200, 500, and 800 Watts for 60 seconds and then washed until a pH of 7 was reached. The activated carbon obtained from this study was found to be a promising material for adsorption applications, as the surface area was improved to a maximum of 400 m²/g using the microwave-assisted method.

The activating agent is one of the most important factors to consider when preparing the activated carbon. Commonly used activating agents include H₃PO₄, ZnCl₂, and KOH due to their environmental friendliness. Among these, KOH is considered an effective alkaline activating agent, as it can enhance the surface area of activated carbon. However, the physical properties and quality of activated carbon also depend on the carbon precursors [23, 24].

Herein, we developed a low-cost activated carbon derived from spent coffee grounds through carbonization without inert gas flow to reduce the preparation cost. The coffee grounds were chemically activated with KOH before undergoing physical activation using the microwave-assist method for Rhodamine B adsorption in aqueous solution. Although the preparation of activated carbon from spent coffee grounds is well established, this study focuses on developing a simple and accessible method to prepare activated carbon from coffee waste that can be performed with basic equipment and proper safety precautions that making it more accessible for a small-scale laboratory or educational setting.

III. MATERIALS AND METHODS

A. Materials and Instruments

Spent coffee grounds were obtained from a local coffee shop in Pathio, Chumphon, Thailand. The chemicals used in this study were of analytical grade. Sulfuric acid (98%) was purchased from QREC, New Zealand. Potassium hydroxide and sodium hydroxide were obtained from KEMAUS, Australia. Rhodamine B was supplied by Loba Chemie Pvt. Ltd., India. The instruments used included a bench pH meter (model STARTER3100) from Ohaus, New Jersey, USA, an ultraviolet-visible spectrophotometer (model V-750) from Jasco, Japan, and a benchtop shaker (model innova®40) from New Brunswick Scientific, New Jersey, USA.

B. Preparation of Adsorbents

The coffee grounds were dried in a hot air oven at 105°C overnight and then washed with 1M H₂SO₄ (v/v) at a ratio of 1g of coffee grounds to 1 mL of H₂SO₄. The coffee grounds were soaked and shaken for 24 h at an agitation speed of 150 rpm and a fixed temperature of 25 °C. After shaking, the materials were washed with DI water and dried in the hot air

oven at 105 °C overnight. The materials were then carbonized at 600 °C for 2 h. The resulting carbon was chemically activated by soaking it in a KOH solution for 24 h at a 1:1 ratio. This was done by mixing 10 grams of carbon with 10 grams of KOH and adding 50 mL of DI water. After KOH activation, the carbon was dried overnight and then physically activated using a microwave-assisted process at a power of 300 watts for a total of 180 s, with a 60-second interval to prevent potential overheating as no inert gas flow was used. The obtained activated carbon was washed several times with DI water until the pH of the rinsed water became neutral. The materials were then dried overnight at 105°C and stored in a desiccator before being used.

C. Batch Experiment for Rhodamine B Adsorption

The adsorption experiments were conducted using a batch method. In each experiment, 20 mg of activated carbon was weighed into an Erlenmeyer flask, and 50 mL of a 15 mg/L Rhodamine B solution was added (equivalent to 0.015 g/L). The mixture was agitated at 150 rpm and a pH of 7 for varying durations from 15 to 150min to evaluate equilibrium conditions. Samples were periodically collected and filtered using Whatman No.1 filter paper. The concentration of Rhodamine B was determined using a UV spectrophotometer at a maximum absorbance wavelength of 554 nm.

The influence of pH on Rhodamine B adsorption was evaluated by weighing 20 mg of activated carbon into an Erlenmeyer flask and adding 50 mL of 15 mg/L Rhodamine B solution. The pH values were varied from 3–11, and they were adjusted using 0.05 M NaOH and HCl before the experiments. The mixtures were shaken at 150 rpm for 15min.

The effect of initial Rhodamine B concentration was investigated by placing 20 mg of activated carbon in an Erlenmeyer flask, followed by the addition of 50 mL of Rhodamine B solutions with concentrations of 3–15 mg/L. The mixtures were shaken at a constant rate of 150 rpm for 15min.

Adsorption isotherms were studied by adding Rhodamine B solutions at concentrations of 3–15 mg/L to 20 mg of activated carbon in an Erlenmeyer flask. The pH of all solutions was adjusted to 7 before the experiment. The mixtures were agitated at 150 rpm or for 15min for further fitting to the adsorption equations.

Kinetics studies were performed by weighing 20 mg of activated carbon into an Erlenmeyer flask, followed by the addition of 50 mL of 15 mg/L of Rhodamine B solution. The pH of Rhodamine B was adjusted to 7, and the shaking speed was set at 150 rpm. Samples were collected at specific time intervals from 15 to 150min for analysis.

IV. RESULT AND DISCUSSION

A. Effect of Acid Washing on Ash Formation

The effect of acid washing on ash formation was evaluated by comparing carbonized samples derived from unwashed and acid-washed raw coffee grounds. As shown in Fig. 1, visible ash deposits were observed in the unwashed sample after carbonization, whereas no ash residue was present in the acid-washed sample. This indicates that acid washing effectively removes ash-forming impurities before carbonization. Our findings are consistent with the study by

Hwang *et al.* [25], who reported that the acid washing of materials before carbonization leads to a lower ash content, contributing to an increase in specific surface area. The acid-treated materials were used as precursors for the next preparation step.

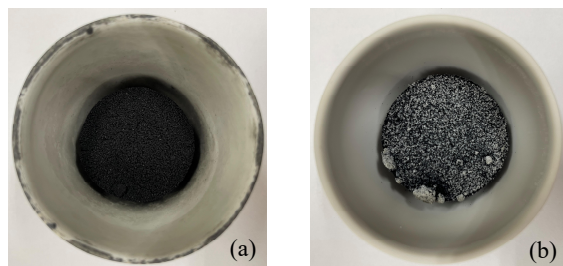


Fig. 1. Effect of acid washing on ash formation (a) washed with 1M H_2SO_4 and (b) unwashed with 1M H_2SO_4 .

B. Characterization of Coffee-Based Activated Carbon

1) Scanning Electron Microscopy (SEM)

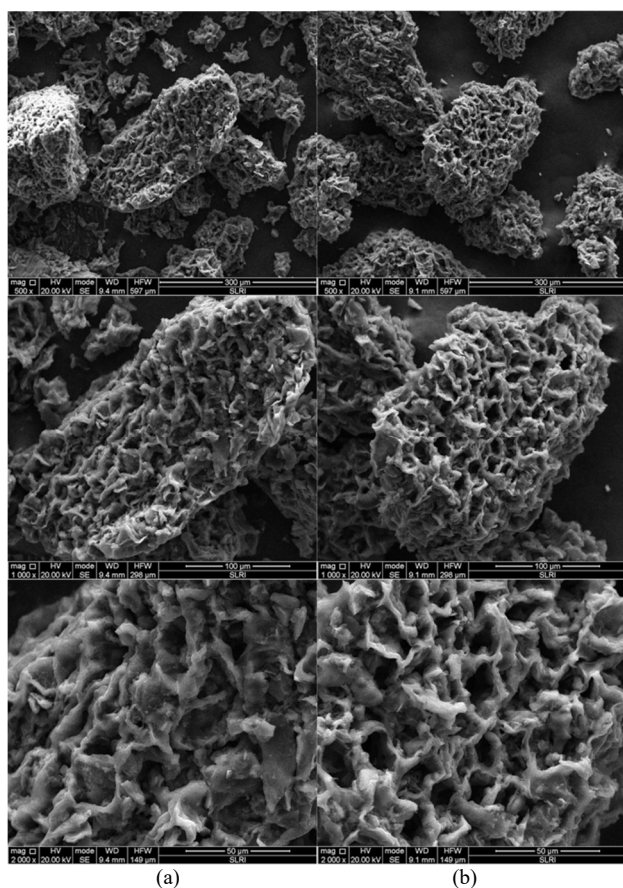


Fig. 2. The SEM images of (a) coffee grounds carbonized at 600 °C for 2 h and (b) coffee grounds carbonized at 600 °C for 2 h, activated with KOH and using a microwave-assisted method.

The SEM images of prepared coffee-based activated carbon at various magnifications are shown in Fig. 2. The first sample was prepared by carbonizing coffee grounds at 600 °C for 2 h without activation, Fig. 2(a). The second sample was subjected to the same carbonization process, followed by KOH chemical activation and microwave-assisted treatment, Fig. 2(b). At 500x magnification, the first sample reveals a relatively compact surface with minimal visible porosity. In contrast, the second sample exhibits a rougher surface with more visible pore formation. At 1000x and 2000x

magnification, it is seen that the second sample has a more open surface structure with more pore openings and well-developed and interconnected pores compared to the first sample. This confirms the successful development of porosity through activation. Previous studies by Liu *et al.* and Indujalekshmi *et al.* [26, 27] reported that KOH activation of carbonized materials commonly leads to the formation of pores due to the reaction of KOH and carbonized materials to form potassium compounds, such as K_2CO_3 and K_2O , which can etch the carbon surface and generate new pores, especially at higher temperatures. While their study used the traditional thermal activation process, the same mechanism applies to our microwave treatment method, where rapid and uniform heating further enhances pore formation.

2) Surface area analysis

The nitrogen adsorption-desorption isotherm of the prepared activated carbon is depicted in Fig. 3. Based on the IUPAC classification, the isotherm corresponds to type IV with an H4 hysteresis loop, which indicates the presence of mesopores and slit-shaped pores within the carbon structure [28–30].

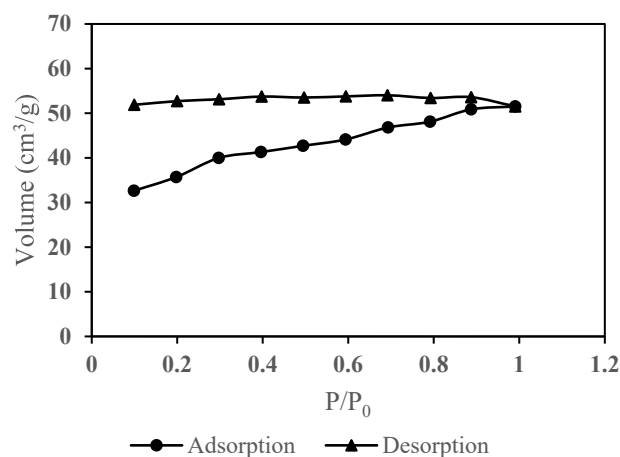


Fig. 3. The nitrogen adsorption-desorption isotherm.

Table 1. Specific surface area and porosity properties of the prepared activated carbon

Parameter	Value
Surface area	120.5 m ² /g
Micropore area	80.36 m ² /g
Total pore volume	0.079 cm ³ /g
Micropore volume	0.039 cm ³ /g
Average pore diameter	2.64 nm

The specific surface area and porosity properties of the prepared activated carbon are shown in Table 1. The Brunauer-Emmett-Teller (BET) analysis revealed that the specific surface area of the prepared coffee-based activated carbon was found to be 120.5 m²/g with a micropore area of 80.36 m²/g. This indicates that micropores dominate the structure. The total pore volume was 0.079 cm³/g, and the micropore volume accounted for 0.039 m³/g. This further supports that the material has microporous characteristics. The average pore diameter was found to be 2.64 nm, which is considered to be in the mesoporous range according to the IUPAC classification [31]. Therefore, the material contains micropores and mesopores in its structure. A previous study by Chun *et al.* [32] reported that KOH activation can create both micropores and mesopores. Moreover, Shi *et al.* [33]

also reported that microwave/KOH activation leads to a more developed pore structure. Similarly, studies by Baytar and İzgi *et al.* [34, 35] demonstrated that the microwave-assisted method significantly improved the surface area of activated carbon derived from agricultural by-products. These studies emphasize the effectiveness of microwave microwave-assisted method in improving the structural properties of activated carbon.

C. Effect of Contact Time

Adsorption equilibrium of Rhodamine B onto activated carbon was monitored over varying time intervals at 15, 30, 60, 90, 120, and 150 min. The amount of Rhodamine B adsorbed per gram of activated carbon (q) at each time interval was calculated using Eq. (1) [30]

$$q_e = \left(\frac{C_0 - C_e}{m} \right) \times V \quad (1)$$

where q_e is the adsorption capacity at equilibrium (mg/g), C_0 is the initial concentration (mg/L), C_e is the concentration at equilibrium (mg/L), V is the volume of Rhodamine B solution (L), and m is the mass of activated carbon used (g)

The effect of contact time on Rhodamine B adsorption is shown in Fig. 4. The results revealed that the rate of adsorption rose sharply in the first 15 min and became constant afterward. The adsorption capacity, calculated using Eq. (1), was found to be 14 mg/g. This indicated that the adsorption reached equilibrium, as most active sites on activated carbon were occupied and no significant changes were observed. Therefore, a contact time of 15 min was chosen as the optimum condition for subsequent experiments.

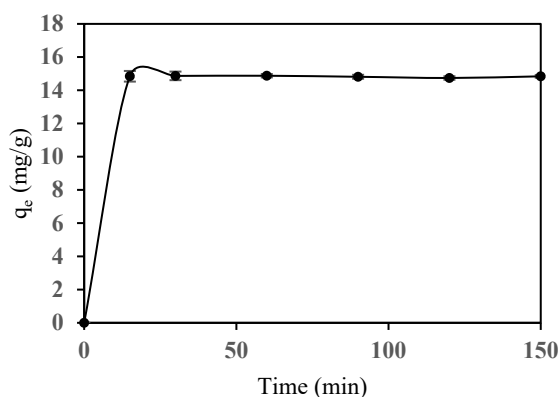


Fig. 4. Effect of contact time.

D. Effect of pH

The influence of pH on Rhodamine B adsorption was investigated using pH at 3, 5, 7, 9, and 11. The effect of pH is illustrated in Fig. 5. The results demonstrated that the adsorption capacity remained relatively stable across the pH range, with only slight fluctuation observed at pH 5 and 9. This indicated that the adsorption of Rhodamine B onto activated carbon was not strongly affected by pH in this range. While many studies have reported that the adsorption of Rhodamine B on activated carbon is pH-dependent, with the adsorption capacity increasing as pH increases. This is due to the change in surface charge. At lower pH, the surface of activated carbon becomes positively charged due to protonation, which leads to the electrostatic repulsion between the positively charged sites on the activated carbon

surface and the positively charged Rhodamine B molecules. In contrast, at higher pH ranges, the negatively charged is predominant on the activated carbon surface, which enhances the electrostatic interaction between the negatively charged surface and cationic Rhodamine B molecules. Therefore, the adsorption capacity becomes higher.

However, in this study, the adsorption appears to occur via non-electrostatic interactions, such as π - π stacking and hydrophobic interactions. These interactions may occur due to the presence of benzene and aromatic rings in Rhodamine B, which can interact with the aromatic structure of activated carbon [30, 36]. For further experiments, pH 7 was chosen because pH 7 represents natural water conditions, which are commonly found in the environment.

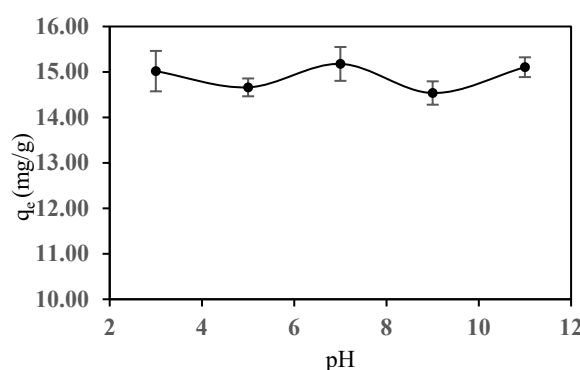


Fig. 5. Effect of pH.

E. Effect of Initial Rhodamine B Concentration

To overcome the mass transfer resistance between the solid phase (activated carbon) and aqueous phase (Rhodamine B solutions), the initial Rhodamine B concentration on adsorption was investigated at 3, 5, 10, and 15 mg/L. Rhodamine B solution was adjusted to pH 7 for each concentration. The adsorption capacity at equilibrium was calculated using Eq. (1). The effect of initial Rhodamine B concentration is depicted in Fig. 6.

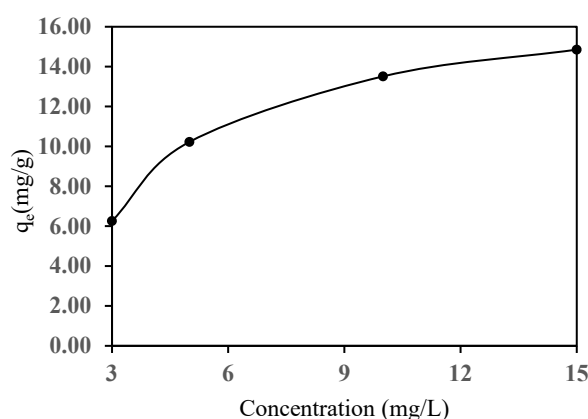


Fig. 6. Effect of initial Rhodamine B concentration.

The results indicated that the adsorption capacity increased with the initial concentration of Rhodamine B, but it appeared to stabilize at higher concentrations. This is due to the limited availability of active sites on the surface of activated carbon and the reduced driving force for mass transfer. At lower concentrations, the active sites of activated carbon are more available, which enhances adsorption efficiency. However, as the surface area of activated carbon becomes limited, the

active sites become saturated, and the ability to adsorb more Rhodamine B decreases. Therefore, at higher concentrations, the mass transfer from the bulk solution to the surface of the adsorbent decreases [36].

F. Adsorption Isotherms

The adsorption isotherms of Rhodamine B on coffee grounds-based activated carbon were performed by varying Rhodamine B concentration at 3, 5, 10, and 15 mg/L. The pH value was adjusted to 7 for each concentration. The results were analyzed using the Langmuir and Freundlich models, which were calculated using Eq. (2) and Eq. (3) [20], respectively. The adsorption isotherms data are represented in Table 2. The Langmuir and Freundlich isotherms plots are shown in Fig. 7.

$$\frac{C_e}{q_e} = \frac{1}{K_L \times q_m} + \frac{1}{q_m} C_e \quad (2)$$

where C_e is the concentration at equilibrium (mg/L), q_e is the adsorption capacity at equilibrium (mg/g), K_L is the Langmuir constant (L/mg), and q_m is the adsorption capacity of the monolayer (mg/g).

$$\log q_e = \log K_F + \frac{1}{n} \times \log C_e \quad (3)$$

where K_F is Freundlich constant (mg/g(L/mg)^{1/n}), and $1/n$ is the adsorbate for the adsorbent.

Table 2. Adsorption isotherms for Rhodamine B adsorption onto activated carbon

Isotherm Models					
Langmuir			Freundlich		
q_m (mg/g)	K_L (L/mg)	R^2	K_f (mg/g(L/mg) ^{1/n})	$1/n$	R^2
16.72	0.73	0.9986	8.91	0.259	0.8538

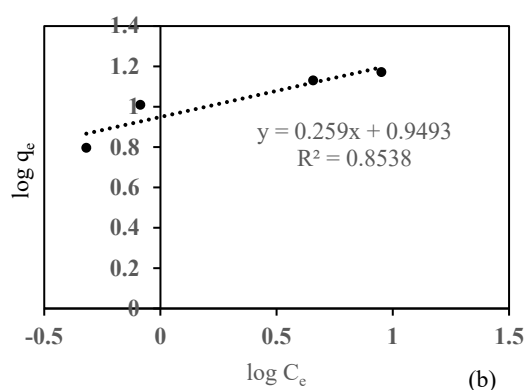
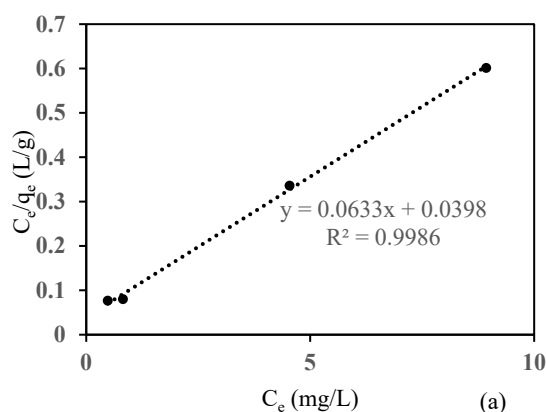


Fig. 7. Adsorption isotherm of RhB on activated carbon: (a) the Langmuir model and (b) the Freundlich model.

According to the data in Table 1 and the plots of Langmuir and Freundlich isotherm in Fig. 3, the results indicate that the adsorption of Rhodamine B onto activated carbon fits the Langmuir model better than the Freundlich model, as the correlation coefficient (R^2) for the Langmuir model is 0.9986, which is higher than that of Freundlich model at 0.8538. This suggests that the adsorption process occurs via a monolayer mechanism on a homogeneous surface. This correlates with the findings of Yin *et al.* for Rhodamine B adsorption [37]. The maximum adsorption capacity (q_m) was found to be 16.72 mg/g, indicating the saturation limit of activated carbon for Rhodamine B in this study. Therefore, the Langmuir model is more suitable for describing the adsorption of Rhodamine B onto activated carbon.

G. Kinetic Studies

The kinetic studies for Rhodamine B onto activated carbon were evaluated using pseudo-first-order and pseudo-second-order models, which can be calculated using Eq. (4) and Eq. (5), respectively [30].

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (4)$$

where q_e is the amount of Rhodamine B adsorbed at equilibrium (mg/g), q_t is the amount of Rhodamine B adsorbed at time t (mg/g), k_1 is the rate constant for the pseudo-first-order model (1/min), and t is the time (min)

$$\frac{t}{q_t} = \frac{1}{K_2 \times q_e^2} + \frac{t}{q_e} \quad (5)$$

where K_2 is the rate constant for the pseudo-second-order model (g/mg-min)

The adsorption kinetics of Rhodamine B onto coffee-grounds-based activated carbon were investigated using both the pseudo-first-order and pseudo-second-order models. The pseudo-first-order models exhibited a rate constant (k_1) of 0.0342 1/min with a relatively low value of R^2 of 0.8132. This indicated that the adsorption might involve a physical process, possibly due to weaker interactions between Rhodamine B and the activated carbon surface. The lower R^2 value also indicated that the adsorption did not fit well with the pseudo-first-order in this study. In contrast, the value of 1.000 of R^2 indicated that the adsorption of Rhodamine B fits better with pseudo-second-order models and follows a chemical adsorption mechanism.

The adsorption mechanism of Rhodamine B onto KOH-activated carbon may occur via both surface chemistry and pore structure. KOH activation enhances the surface area and introduces carboxyl groups on the activated carbon surface, which contribute to strong electrostatic interactions with the cationic Rhodamine B molecules. This chemisorption mechanism is consistent with the pseudo-second order kinetics, which indicates that the rate of adsorption is determined by the formation of chemical bonds between the dye and the adsorbent surface. The mechanism involves the diffusion of dye molecules to the surface, penetration into the pores, and binding at active sites. This adsorption mechanism aligns with the report by Indujalekshmi *et al.* [27], where KOH activation was found to improve adsorption through surface functionalization and increased surface area, leading to an enhancement in the interaction between the Rhodamine B and adsorbent. The summary of adsorption kinetics for

Rhodamine B adsorption is shown in Table 3. The adsorption kinetics of Rhodamine B on activated carbon are depicted in Fig. 8.

Table 3. Adsorption kinetics for Rhodamine B adsorption onto activated carbon

Kinetic Model	k_1 (1/min)/ k_2 (g/mg·min)	q_e (mg/g)	R^2
Pseudo-first-Order	$k_1 = 0.0342$	2.66	0.8132
Pseudo-second-Order	$k_2 = 0.579$	126.58	1.0000

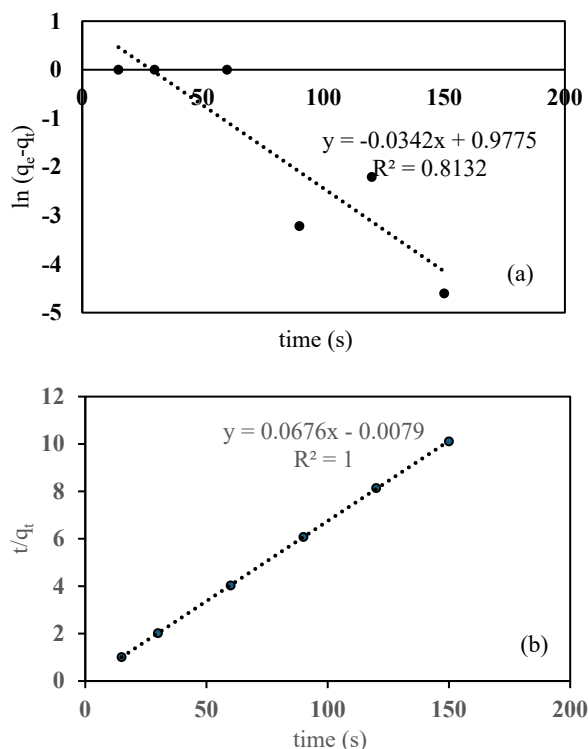


Fig. 8. Adsorption kinetics of methylene blue on activated carbon (a) pseudo-first-order and (b) pseudo-second-order.

V. CONCLUSION

This study demonstrated the successful preparation of low-cost activated carbon from coffee grounds for the removal of Rhodamine B. The activation process was carried out without the need for an inert gas flow during both the carbonization and microwave-assisted activation steps, which makes it more practical and cost-effective. The adsorption kinetics follow the pseudo-second-order model provided, indicating that the adsorption process is chemically controlled. The adsorption data fitted well with the Langmuir isotherm model, which indicated that the adsorption occurs via a monolayer adsorption mechanism. This activated carbon has potential for further development in a wide range of adsorption applications.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Kanchalar Keeratirawee conceptualized and designed the study, conducted the experiments, analyzed the data, and wrote the manuscript. Orasa Choolaaied prepared the chemical used and conducted the experiments. Pornprapa Kongtragoul provided sources, assisted with the data analysis;

all authors had approved the final version.

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