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Kinetic Analysis of Methylane Blue Adsorption using Cocoa Shell Biochar (Theobroma Cacao L) With a Combination of Chemical and Physical Activators

Galang Ariyuda[1*], Vera Listyawati[2], Retno Dwi Nyamiati[3]
Universitas Pembangunan Nasional "Veteran" Yogyakarta
Corresponding Author: Galang Ariyuda ariyudagalang@gmail.com

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ABSTRACT

The textile industry is a major contributor to severe global water pollution, releasing pollutants like dyes, heavy metals, and pesticides. A practical solution for removal is adsorption. This study successfully utilized cocoa pod husk, an agricultural waste, as a low-cost adsorbent. The husk was converted into biochar via pyrolysis and chemically activated with 0.1 M HNO3. A microwave was used for drying the activated biochar. The kinetic tests identified the optimal conditions for adsorption as a 0.01 M NaOH concentration, pH 3, a methyl orange (MO) concentration of 70 mg/L, and a temperature of 25°C. The process was determined to be a pseudo-second order reaction. This approach offers a dual benefit: effectively treating polluted water and valorizing cocoa farming waste.

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INTRODUCTION

Textiles are a basic human need that has existed since ancient times, reflecting how humans have strived to meet clothing needs since the dawn of civilization. Initially, textiles served only to protect the body from the weather and the environment, but as civilization developed, textiles took on a broader role. Beyond just covering the body, textiles also became symbols of social status, cultural expression, and the identity of a community. The diverse motifs, colors, and materials of textiles reflect the richness of tradition and creativity in each region, from batik in Indonesia to silk in China. To meet increasingly complex needs, the textile industry has experienced rapid development, both in terms of technology, material innovation, and design. Increasing market demand has driven the emergence of new technologies such as synthetic fibers, environmentally friendly dyes, and more efficient production techniques, making textiles a strategic sector in the global economy.(Graph & Spectral, 2022).

However, behind the rapid development of the textile industry lies significant challenges related to its environmental impact. One of the most serious issues is the waste generated during the production process, particularly during the dyeing and dyeing stages. This process produces large amounts of liquid waste that often contains hazardous pollutants such as synthetic dyes, toxic chemicals, and heavy metals. These substances can damage ecosystems if disposed of without adequate treatment, polluting rivers, lakes, and even groundwater sources that are vital for humans and other living things. Furthermore, the enormous use of water in production increases pressure on the availability of clean water in various regions. This situation has sparked global concern and demands the implementation of more sustainable solutions, such as environmentally friendly waste management technologies, the implementation of circular economy principles, and innovations in safer and more biodegradable textile materials.(Made Wipra Pratistita et al., 2024).

One of the synthetic dyes that is often used in the textile industry is methylene blue.(Maryudi et al., 2021) Methylene blue is a synthetic dye known for its bright blue color, often used in dyeing denim, silk, and various other textile materials. Its strong and long-lasting color makes it a top choice in the textile industry. However, the use of methylene blue also carries environmental and health risks if waste containing this substance is disposed of without adequate treatment. When methylene blue waste pollutes the air, its distinctive color can disrupt aquatic ecosystems. The resulting blue color can block sunlight from penetrating the air, thus disrupting the photosynthesis process of aerial plants. This can reduce dissolved oxygen levels, which negatively impacts aquatic life. Furthermore, water contamination by methylene blue can affect drinking water quality and the sustainability of clean water sources. From a human health perspective, long-term exposure to methylene blue through direct contact or consumption of contaminated air can cause various problems, such as irritation of the skin, eyes, and respiratory tract. In certain cases, this substance can also trigger allergic reactions or other complications, especially in individuals who are sensitive to certain chemicals. Not only does methylene blue pollute the air, it can also damage soil quality. If textile waste containing this substance seeps into the soil, the presence of methylene blue can disrupt the chemical structure of the soil and affect fertility. As a result, plant growth is inhibited, which can impact agricultural productivity in affected areas. To address this problem, it is important for the textile industry to implement more effective wastewater treatment technologies. Methods such as adsorption using natural materials, photocatalysis using UV light, or biological treatment are essential.(Oladoye et al., 2022).

To address the problem of textile waste pollution, various waste treatment methods have been developed to minimize its negative impact on the environment. One promising method is adsorption using biochar. Biochar is a porous carbon material produced through pyrolysis, the heating of organic matter at high temperatures in the absence or minimal oxygen.

The advantages of biochar lie in its large surface area, complex pore structure, and high adsorption capacity. These properties make biochar a highly effective material for adsorbing various types of pollutants, including synthetic dyes such as methylene blue, which are often found in textile waste. The adsorption process using biochar is not only efficient in removing color from wastewater but can also reduce the concentration of toxic chemicals, heavy metals, and other organic compounds. Furthermore, the use of biochar also has added value in terms of desirability. Biochar can be made from agricultural waste, wood, or other unused biomass, thus supporting the principles of a circular economy and integrated waste management. The production process produces a byproduct in the form of usable heat energy, making it a multifunctional, environmentally friendly technology. In textile wastewater treatment applications, biochar can be used in adsorption reactors or filtration columns to clean wastewater before it is discharged into the environment. Its effectiveness in reducing pollutant concentrations has been demonstrated in various studies. However, challenges such as biochar regeneration, used biochar management, and production costs still need to be optimized to increase its scalability and competitiveness. With further development, adsorption using biochar has the potential to become the main solution in addressing textile waste pollution sustainably.(Lonappan et al., 2016).

Cocoa husk is an abundant agricultural waste. By processing cocoa husk into biochar, we can utilize this waste into a value-added product while addressing environmental concerns. Cocoa husk biochar has great potential as an adsorbent for removing methylene blue from textile wastewater. This is due to the presence of functional groups on the biochar surface that can interact with methylene blue molecules through physical and chemical adsorption mechanisms.(Nurhayati et al., 2021).

LITERATURE REVIEW

Adsorption

Adsorption is the process of separating components from a fluid by transferring them to the surface of a solid adsorbent. This occurs due to molecular differences, which cause stronger surface binding for some molecules. The process requires an adsorbent, an adsorbate, and time to reach equilibrium. There are two main types: chemical adsorption (chemisorption) and physical adsorption (physisorption). Chemisorption involves strong chemical bonds and reactions, forming new compounds, and is often difficult to reverse. In contrast, physisorption is driven by weaker physical forces.(Putri et al., 2022).

Microwaves

Microwaves are electromagnetic radiation with wavelengths ranging from 1 mm to 1 m and frequencies between 300 MHz and 300 GHz. In industrial applications, the most commonly used frequencies for heating purposes are 915 MHz, 2.45 GHz, 5.8 GHz, and 22.125 GHz (Ewis & Hameed, 2021). Microwave technology has been used in materials science and processing, including powder synthesis and polymer synthesis (Sosnik et al., 2011). Specifically, microwave technology as a heating source has been used in adsorbent synthesis compared to conventional heating because of: low energy consumption and processing time fast and simple irradiation process uniform temperature throughout the material improved diffusion process.(Dharmalingam et al., 2018).

Reaction Order

Adsorption During the adsorption process, the concentration of adsorbate will decrease over time. The number of concentration factors that affect the reaction rate is also called the reaction order. Apparent first-order kinetics, apparent second-order kinetics, Elovich kinetics, and SS intraparticle diffusion kinetics are expressed in the following equations, respectively:

$$q_t = q_e(1 - e^{-k_1 t}) \quad (1)$$

$$\frac{t}{q_t} = \frac{1}{k_2\, q_c^2} + \frac{t}{q_c} \quad (2)$$

$$q_t = \frac{\ln a_e b_e}{b_e} + \frac{1}{b_e}\ln t \quad (3)$$

$$qt = k_{ip}t^{0,5} + C \quad (4)$$

Where q_t (mg/g) is the adsorption capacity of an adsorbate by an adsorbent at time t, while q_e (mg/g) indicates the adsorption capacity at equilibrium, k 1 (1/h) and k 2 [g/(mg ·h)] are the kinetic constants of the pseudo-first-order model and pseudo-second-order model. k ip [mg/(g ·h 0.5)] is the diffusion rate within the particle, C is a constant related to the thickness of the boundary layer. The equation with the highest R^2 is selected as the most appropriate order kinetics.(Huang et al., 2023a).

METHODOLOGY

Subject

In this study, chemical and physical activation will be carried out. Chemical activation is carried out by activating cocoa shell charcoal which has previously been pyrolyzed at a temperature of 340 C for 3 hours and ground to 100 mesh with 0.01 M NaOH activator, and physical activation using a microwave.

Charcoal preparation stage

Pyrolysis is a thermochemical conversion process that converts biomass or organic material into three main types of products: solids (biochar), liquids (bio-oil), and gases (pyro-gas), through heating in the absence of oxygen. Operating conditions in pyrolysis greatly affect the physicochemical properties, quality, and yield of the products produced. Therefore, process parameters can be modified to optimize pyrolysis product yields, both in terms of quality and quantity. Due to its simplicity, ease of implementation, and potential for large-scale application, pyrolysis has become the focus of much research, particularly in the production of bio-oil from biomass. Meanwhile, biochar is often considered a byproduct that can be utilized as an energy source through combustion.(Al-Rumaihi et al., 2022). Cocoa shells are cleaned and chopped into 10-30 mm pieces and dried in direct sunlight for 2-3 days, then dried in an oven for 4 hours at 105°C to reduce the moisture content to <10%. A total of 3 kg of dried cocoa shells are placed in a pyrolysis reactor. The pyrolysis temperature is 340°C. After 3 hours, the cocoa shell residue is removed, dried, and crushed into powder. Next, the crushed cocoa shells in powder form are sieved to a size of 100 mesh.

Chemical and physical activation processes

During the adsorbent activation process, chemical and physical activation are carried out. The result of the pyrolysis process, which is cocoa shell charcoal, is chemically activated using NaOH at a ratio of 1:7 (activated carbon (1 g) and HNO_3 (7 ml)). Next, the charcoal is placed in an Erlenmeyer flask with a concentration of 0.1 M HNO_3 for 4 hours. After 4 hours, it is neutralized by adding distilled water until the pH is neutral (=7). This physical carbon activation process uses a 500-watt microwave oven at a temperature of 100 °C for 10 minutes.

Analysis PHpzc

The preparation of the PHpzc stock solution uses a 1 M KCL solution of 74.55 g/l, which is homogenized with distilled water. It is placed in an Erlenmeyer flask with each of the following pH variations: 6.7, 8, 9, and 10. Next, 0.1 grams of activated carbon is added to each pH variation and shaken for 24 hours. After 24 hours, the filtrate was measured using a pH meter. A line was plotted between the initial pH and the pH delta on a graph, and the intersection was obtained at y = 0.

Adsorption Processes

Overall, adsorption is a process in which certain components are separated from a solution in a fluid phase and transferred to the surface of a solid substance that has the ability to absorb, called an adsorbent. This separation process occurs due to differences in molecular weight or porosity, which cause some molecules to bond more strongly to the surface than others. The adsorption capacity of an adsorbent can be calculated using the formula:

$$q_e = \frac{(C_0 - C_e)V}{M}$$

$$q_t = \frac{(C_0 - C_t)V}{M}$$

(Huang et al., 2023a)

$$\%\text{removal} = \frac{C_0 - C_e}{C_0} \times 100$$

(Serban et al., 2023)

Description:

q_e = amount of adsorbate adsorbed at equilibrium.
C_0 = initial adsorbate concentration.
C_e = adsorbate concentration at equilibrium.
V = adsorbate solution volume.
M = adsorbent mass used.
q_t = amount of adsorbate adsorbed at time t.
C_t = adsorbate concentration at time t.

Prepare a 100 mg/l methylene blue stock solution of 100 ml. Prepare 25 ml of methylene blue solution with a concentration of 50 mg/l in 6 bottles with pH variations of 6, 7, 8, 9, and 10. Take 10 ml of each solution and put it in an Erlenmeyer flask, then add 0.1 g of activated carbon and shake at a speed of 150 rpm for 60 minutes. Filter the activated carbon and test the concentration of the filtrate obtained with the methyl orange solution that has not been contacted with activated carbon using a spectrophotometer. Calculate the adsorption capacity for each variation. The pH that produces the highest adsorption capacity will be used in the next optimization.

Prepare a 100 mg/l methylene blue stock solution of 100 ml. Prepare 6 x 25 ml methylene blue solutions with an optimum pH and varying concentrations of 50, 60, 70, 80, and 90 mg/l. Take 10 ml of each solution and put it into an Erlenmeyer flask, then add 0.1 g of activated carbon and shake at a speed of 150 rpm for 60 minutes. Filter the activated carbon and then test the concentration of the filtrate obtained using a spectrometer. Calculate the adsorption capacity for each variation. The concentration that produces the highest adsorption capacity will be used in the next optimization.

Prepare a 100 mg/l orange stock solution of 100 ml. Prepare 6 orange solutions with an optimum pH and optimum concentration of 25 ml. Take 10 ml of each solution and put it in an Erlenmeyer flask, then add 0.1 g of activated carbon and shake it at a speed of 150 rpm with contact times varying from 15, 30, 45, 60, to 75 minutes. Filter the activated carbon, then test the concentration of the filtrate obtained using a spectrometer. Calculate the adsorption capacity for each variation. The contact time that produces the highest adsorption capacity will be used in subsequent tests.

Kinetic study

Reaction kinetics or reaction rate is measured from changes in reactant or product concentration over time. Therefore, the kinetics of the adsorption process can be obtained from the rate of decrease in adsorbate concentration over time.

In the adsorption process over time, the concentration of the adsorbate will decrease. The number of concentration factors that affect the reaction rate is also called the reaction order. First-order and second-order kinetics are expressed in the following equations:

$$q_t = q_e\left(1 - e^{-k_1 t}\right)$$

$$\frac{t}{q_t} = \frac{1}{k_2\, q_c^2} + \frac{t}{q_c}$$

Where q_t (mg/g) is the adsorption capacity of an adsorbate by an adsorbent at time t, while q_e(mg/g) indicates the adsorption capacity at equilibrium, k_1 (1/jam) and k_2 (g/(mg/h) are the kinetic constants of the pseudo-first-order model and pseudo-second-order model.(Huang et al., 2023b).

RESEARCH RESULT AND DISCUSSION

pH pzc

The results of determining the pzc pH with initial pH variations of 6, 7, 8, 9, and 10 obtained an initial pH value that intersected the line y = 0, precisely at pH 7.5.

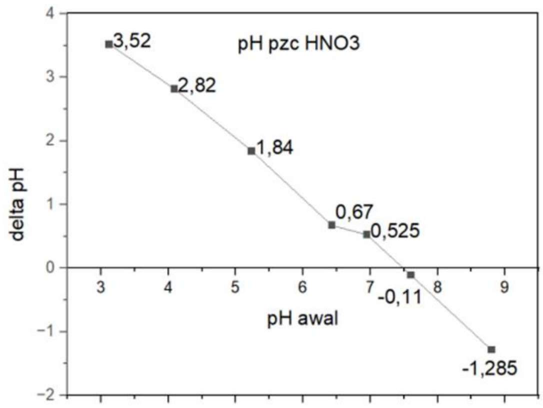

Figure. 1 pH pzc

Optimization

From the entire series of studies conducted to optimize methyl orange adsorption, the adsorption capacity at various pH variations is shown in Table 1.

Table. 1 Optimization

pH	%removal
6	97.891%
7	97.360%
8	99.572%
9	99.598%
10	99.012%

From the table, it can be seen that the optimum pH for cocoa shell biochar for methylene blue adsorption reaches its peak adsorption level at pH 9, with an adsorption amount of 4.894 mg/l and a removal rate of nearly 99.6%. This is the peak adsorption of methylene blue from a pH variation of 6-10, because at pH 10 there is a decrease in the amount of adsorption, namely 4.950 mg/l, which is smaller than at pH 9. This is because at pH 9, methylene blue tends to be in an optimally ionized form, which allows for optimal interaction with the adsorption material. However, at pH 10, there is a decrease in adsorption capacity due to the possibility of degradation or chemical transformation of methylene blue. At high pH, the surface of activated biochar tends to become more negatively charged.(Banerjee et al., 2014)

After determining that pH 9 was the optimum pH, it was then used to optimize the initial concentration of methylene blue. The results are shown in Table 2.

Table. 2 The Results Optimization

Initial concentration	%removal
400	98.24%
700	98.41%
1000	98.95%
1200	99.09%
1400	99.15%
1800	94.24%
2000	89.71%

Based on the data and graph above, the experiment to find the optimum concentration of Methylen blue was carried out up to a concentration variation of 2000 mg/L. This is because in the initial experiment up to a variation of 1200 mg/L, there was still an increase up to 2000 mg/L. When viewed from the results, the concentration of Methylen blue will experience a constant increase in adsorption capacity. This may be due to the phenomenon of multi-layer adsorption, where Methylen blue molecules accumulate on top of each other on the surface of biochar. This can occur because there is still empty space available on the surface of biochar that can accommodate more Methylen blue molecules.

For example, research conducted by (Pan et al., 2025) shows that biochar has a high adsorption capacity for methylene blue, and the adsorption capacity increases with increasing methylene blue concentration until it reaches a certain concentration where the adsorption capacity begins to saturate. Therefore, in this case, the optimum concentration used is 1400 mg/L, which is 138.8142. This is because when viewed in terms of % removal at a concentration of 1400 mg/L, it has reached 99.15%. After this variation of 1400 mg/L, the % removal decreases, indicating that it has reached saturation point. However, a phenomenon of multi-layer adsorption occurs.

Table. 3 The Optimum Time for the Adsorption of Methylene Blue By Cocoa Shell Biochar

Contact time	%removal
15	94.19%
30	97.75%
45	99.10%
60	99.11%
75	98.89%

Based on the data above, it can be seen that the optimum time for the adsorption of Methylene blue by cocoa shell biochar is 45 minutes, with an adsorbed amount of 178.3934 and a removal rate of 99%. This is due to a number of factors involving the interaction between biochar and methylene blue dye, including changes in the structure of biochar, where prolonged contact with methylene blue can damage the structure of biochar itself. In addition, the kinetics of absorption also influence this. Most likely, at the beginning of the experiment, rapid absorption of methylene blue occurred, which was related to the diffusion and adsorption processes. Over time, the absorption capacity of biochar towards methylene blue may become saturated, resulting in a decrease in the absorption rate at 60 minutes and 75 minutes. (Mahanta et al., 2022)

Kinetic study

Kinetic analysis was performed by applying pseudo-first-order kinetic models and pseudo-second-order kinetic models. Kinetic model identification was performed by matching curves connecting time (t) with -ln (qe-qt) for pseudo-first order, and time (t) with t/qt for pseudo-second order. Parameters such as k1, k2, qe, and R^2 from both kinetic models were obtained through the curve linearization process. Illustrations of the curves for both kinetic models can be seen in Figures 2 and 3.

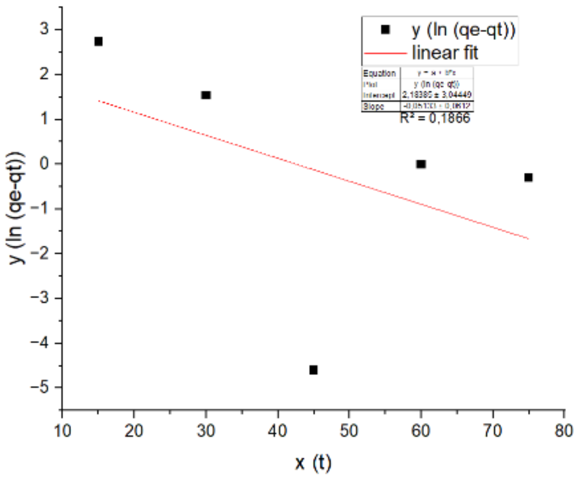

Figure. 2 First-Order Kinetic

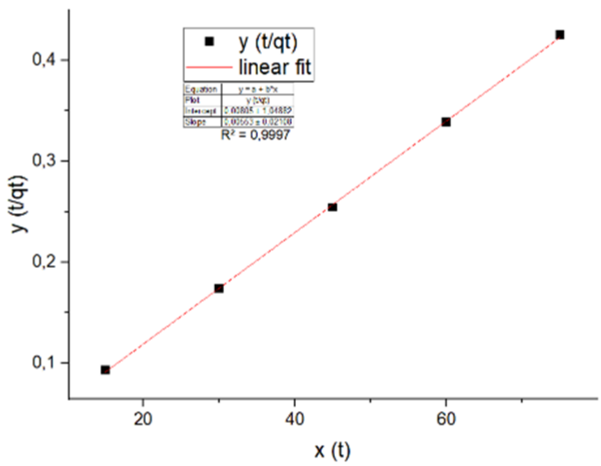

Figure. 3 Second-Order Kinetic

Based on the data in Figures 2 and 3, the coefficient of determination (R^2) for the first pseudo order is 0.1866 with K_1 equal to 0.0508. Meanwhile, for the second pseudo order, the coefficient of determination (R^2) is 0.9997 with K_1 equal to 0.00398. This shows that the kinetic order suitable for methylene blue is the second order, because it has a higher coefficient of determination than the first order.

Second-order kinetics usually occur when adsorption occurs through multilayer interactions between the adsorbate (methylene blue) and the adsorbent (cocoa shell), and when there is more than one mechanism contributing to the adsorption process. In this case, the adsorption process of methylene blue by cocoa shells may involve complex interactions between the dye and various active components in the pore structure of cocoa shells.(Hassan et al., 2018).

CONCLUSIONS AND RECOMMENDATIONS

This study shows that biochar from cocoa shells, which is chemically and physically activated, has high potential as an adsorbent for removing methylene blue from textile waste. By utilizing abundant agricultural waste, such as cocoa shells, this process not only reduces environmental pollution but also provides a sustainable solution for waste management.

The results of the study found that the optimal conditions for methylene blue adsorption were at pH 9, an initial concentration of 1400 mg/L, and a contact time of 45 minutes, with an adsorption efficiency of up to 99.15%. Kinetic analysis showed that this adsorption process followed a pseudo-second-order kinetic model, which describes the multilayer interaction between methylene blue and biochar.

Thus, the use of cocoa shell biochar as an adsorbent offers an environmentally friendly, effective, and economical alternative to address textile waste pollution issues, while supporting the principles of the circular economy through the utilization of biomass waste.

ADVANCED RESEARCH

Still conducting further research to find out more about Kinetic Analysis of Methylane Blue Adsorption using Cocoa Shell Biochar (Theobroma Cacao L) With a Combination of Chemical and Physical Activators.

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