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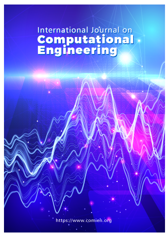

Coconut Shell Waste as an Adsorbent for Methylene Blue Dye Removal

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***Abstract*— Coconut shells, a byproduct with significant economic value, pose environmental challenges when not properly processed. One effective way to mitigate these impacts is by converting coconut shells into activated carbon, which is widely used as an adsorbent due to its superior adsorption capabilities. This study aims to evaluate the quality of adsorbents activated through physical and physico-chemical methods. The parameters assessed include moisture content, ash content, iodine absorption, and methylene blue absorption, all in accordance with the SNI 1995 standards. Additionally, the study investigates the impact of adsorbent mass and contact time on the effectiveness and capacity of coconut shell adsorbents for methylene blue solutions.The research methodology comprises three main stages: adsorbent preparation, adsorbent activation, and the adsorption process. During the adsorption process, various adsorbent masses (5, 10, 15, 20, and 25 grams) and contact times (15, 30, 45, 60, and 75 minutes) are tested using two types of activation: physical and physico-chemical. The results demonstrate that the highest adsorption percentage, 99.91%, is achieved with a physico-chemically activated adsorbent mass of 25 grams and a contact time of 60 minutes. This study underscores the effectiveness of physico-chemical activation and optimal adsorbent mass and contact time in enhancing the adsorption capacity of coconut shell-derived activated carbon for methylene blue solutions. By optimizing these parameters, the environmental impact of coconut shells can be significantly reduced while maximizing their economic value.**

***Keywords*— Adsorption; coconut shell; methylene blue; physical activation; physico - chemical activation; adsorbent mass.**

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I. Introduction

The traditional woven fabric industry in South Sumatra is one of the industrial activities that is one of the main contributors to economic activities in the city of Palembang. Although this industry has promising potential, the waste generated from this dyeing process can cause problems for the surrounding environment.

Waste with a concentrated color and high levels of chemical oxygen demand (COD) will poison life in waters because of the molecular nature of compounds in it that can bind metal ions. Methylen blue is a thiazine dye that is often used because it is easy to obtain and cheap. Methylene blue is one of the dyes in fabric dyeing [1]. Methylene blue is one of the dyes that dissolve in water. High doses of methylen blue can cause nausea, vomiting, abdominal and chest pain, headaches, excessive sweating, and hypertension. One of the compounds that is widely used in the fabric, leather and printing dyeing industry is methylene blue [7]. In addition, Methylen blue can cause irritation of the digestive tract if ingested, cause cyanosis if inhaled, and irritation of the skin if touched by the skin [11].

A. Literature Review

Coconut plants (Cocos Nucifera L) are abundantly found in Indonesia, a tropical country suitable for their growth. These plants belong to the Palmae family and thrive in environments with adequate sunlight, temperature, rainfall, humidity, and soil conditions [5]. One of the most critical parts of the coconut plant is the fruit, consisting of epicarp, mesocarp, endocarp, and endosperm. The epicarp is the smooth, hard outer skin, while the mesocarp is the fibrous middle layer called coir. The endocarp is the hard shell, and the endosperm is the edible inner layer. Despite the widespread use of coconut meat and water in daily life, the coconut shell is often discarded, leading to waste. Utilizing coconut shells can increase their value and reduce waste.

Coconut Coconut shell possesses remarkable properties such as carbon-rich and environmentally friendly solid fuel to other biomass and coal materials; hence, it is possible to produce alternative energy from coconut shell biomass due to its several characteristics [17].

The chemical composition of coconut shells includes cellulose (34%), lignin (27%), hemicellulose (21%), and ash (18%) . This composition makes coconut shells a potential raw material for various applications, including the production of activated charcoal [14].

1) Activated Coconut Shells Charcoal Capability

Activated carbon is a porous solid containing 85-95% carbon, produced from carbon-containing materials heated at high temperatures [9]. Activated carbon is an amorphous compound with a very large surface area, namely 200-2000 m2/g [8]. Activated carbon is often used as an adsorbent during adsorption, because activated carbon has better adsorption power than other adsorbents.

Charcoal is a black residue containing impure carbon that is produced by removing water and volatile components from animals or plants. Charcoal is generally obtained by heating wood, sugar, bones and other objects. This black, lightweight, easily crushed, and coal-like charcoal consists of 85-98% carbon, the rest is ash or other chemical substances [4].

Activated charcoal has a high adsorption capacity, making it effective for removing certain gases and chemicals depending on the pore size and surface area. Activated charcoal is obtained through pyrolysis and subsequent activation using gases like CO^2 or steam, which opens up the pores, increasing its adsorption capacity . The resulting material contains 5-15% water, 2-3% ash, and the rest is carbon. Its high surface area, ranging from 100 to over 3000 m^2/g, makes it suitable for applications in adsorption, catalysis, and reactions.

The quality of activated charcoal is influenced by the raw materials, activators, and processing methods. High-quality activated charcoal has high carbon content, low ash, and low moisture. The Indonesian National Standard (SNI) for activated charcoal specifies a maximum moisture content of 15%, a maximum ash content of 10%, a minimum iodine absorption of 750 mg/g, and a minimum methylene blue absorption of 120 mg/g [15].

Coconut-shell charcoal was found to be comparable to so-called hard charcoal in strength and low ash content, and can also be a reliable source of carbon for the manufacture of chemicals with various purpose [19].

2) Methylene Blue Characteristc

Dyes are widely used in food, cosmetic, pharmaceutical, printing, textile, and leather industries during manufacturing process. As a result, a considerable amount of undesired coloured effluents is generated. The presence of dyes and pigments in water resources not only renders them aesthetically unacceptable, but also poses serious health-risk factors on living organisms and the environment [16].

Methylene blue ($C_{16}H_{18}ClN_3S$) is an aromatic heterocyclic chemical compound with a molecular weight of 319.89 g/mol and a melting point of 105°C. It is commonly used as a dye in textiles, paper, and cosmetics. This dye have a form of dark green crystal at room temperature, if dissolved with water or alcohol and also in an environment with a high level of oxidation, it will turn to dark blue color. This dye is a carcinogen so it is dangerous if it comes into contact with the skin, eyes, and ingestion [3].

Compared with other treatment methods, the adsorption method is considered as prevailing over other dye wastewater treatment technology due to its advantages such as high efficiency, low cost, simple operation, and insensitive of toxic substances such as Methylene Blue. Activated carbon is the most commonly used adsorbent, and is widely used to remove the organic and inorganic pollutants in water phase [20].

3) Adsorption Process

Adsorption is the process of coagulation of dissolved substances in a solution by the surface of an absorbent that makes the ingress of the substance and accumulates in an absorbent substance. Both often appear at the same time as a process, so some call it asorption. In Adsorption, there are things called Adsorbents and Adsorbents. Adsorbents are absorbent substances, while adsorbates are absorbed substances [2].

Adsorption involves the accumulation of substances from a liquid onto a solid surface. Adsorbents, such as activated charcoal, have pores that increase their surface area, facilitating this process. The effectiveness of adsorption depends on factors like contact time, adsorbent mass, agitation, and temperature .

Among several agricultural wastes studied as biosorbents for water treatment, coconut has been of great importance as various parts of this tree (e.g. coir, shell, etc.) have been extensively studied as biosorbents for the removal of diverse type of pollutants from water. Coconut-based agricultural wastes have gained wide attention as effective biosorbents due to low-cost and significant adsorption potential for the removal of various aquatic pollutants [18].

Adsorption can occur through physical or chemical means. Physical adsorption involves weaker Van der Waals forces, while chemical adsorption involves stronger covalent bonds between the adsorbent and the adsorbate. Several factors influence the adsorption capacity, including; **Contact Time:** Longer contact times allow for more adsorption until equilibrium is reached. The more collisions that occur, the faster the reaction takes place until equilibrium conditions reached [6]., **Adsorbent Mass:** Increasing the adsorbent mass increases adsorption sites, enhancing capacity., **Agitation:** Agitation ensures better contact between the adsorbent and adsorbate., **Temperature:** Adsorption is typically exothermic, meaning lower temperatures enhance the process .

Adsorption can be performed using static (batch) or dynamic (column) methods. The static method involves mixing the adsorbent with the solution and separating them after a specific time. In contrast, the dynamic method involves passing the solution through a column containing the adsorbent .

The adsorption ability of activated carbon depended on the pore size distribution which was related to the adsorbate trapping and the capability of the adsorbent to retain the adsorbate [21]

B. Research Objective

The purpose of this study are producing coconut shell adsorbent which is activated physically and chemically with parameters of water content, ash content and iodine absorption capacity according to SNI standards and analyze the effect of physically activated adsorbent mass as well as physical chemical and contact time on the effectiveness of coconut shell adsorbent (against methylene blue solution)

II. Materials and Methods

The research was conducted at the Laboratory of the Chemical Engineering Study Program, Muhammadiyah University of Palembang. The research period was from April to July 2023.

A. Preparation of Activated Carbon from Coconut Shells

The coconut shells were first thoroughly cleaned and then sun-dried for 2-3 days. After dried, it were placed in a container and subjected to carbonization at 300°C for 1 hour to form charcoal. The charcoal obtained from the coconut shells was then ground and sieved using a 100 mesh sieve. After the sieving process, the charcoal was further heated in a furnace at 500°C for 1 hour, and the final product shall be stored in a dessicator for the upcoming treatment.

Fig. 1 The crushed shells before (A) & after (B) sifted using a 100 mesh sieve.

B. Physcal-Chemical Activation

The physically activated coconut shell charcoal was soaked in a 0.5 M phosphoric acid (H_3PO_4) solution, stirred, and covered for 24 hours. After the immersion process, the charcoal was then washed with distilled water until the pH was neutral. The washed charcoal was dried in an oven at 100°C for 1 hour. and the results stored in a dessicator for the upcoming experimental steps.

C. Adsorption Process

To investigate the adsorption efficiency of activated carbon derived from coconut shells for the removal of methylene blue, a series of experiments were conducted with both physically and physically-chemically activated carbon.

For the physically and physically-chemically activated carbon, ten samples of methylene blue solution each for the two different type of adsorbent, each with a concentration of 20 ppm and a volume of 50 ml, were prepared. Different masses of adsorbent (5, 10, 15, 20, and 25 grams) were added to each sample. The mixtures were stirred at a constant speed of 130 rpm for 60 minutes. After stirring, the solutions were filtered using filter paper, and the resulting filtrates were analyzed with a UV-Vis spectrophotometer to determine the remaining concentration of methylene blue.

Next, the effect of contact time was studied using the optimal adsorbent mass determined from the previous experiment. Five samples of methylene blue solution (20 ppm, 50 ml each) were prepared, and the optimal mass of adsorbent was added to each. The contact times tested were 15, 30, 45, 60, and 75 minutes, with stirring at 130 rpm. After the designated contact times, the solutions were filtered and analyzed with a UV-Vis spectrophotometer.

These procedures allowed for the assessment of the adsorption efficiency of both physically and physically-chemically activated carbon under different conditions, providing valuable insights into the optimal parameters for the removal of methylene blue from aqueous solutions.

Fig. 2 Mixing the adsorbent with a 20 ppm methylene blue solution.

D. Analytical Methods

All parameters in this study were conducted in triplicates and followed: Ash content was measured according to AOAC official method 942.05 (AOAC, 2012), water content were measured by the weight before and after the sample inside an oven[12]. Iodium Absorption Analysis & Absorbency to methylene blue was measured with SNI, 1995 using titration methods [13]. And the last step was to calculate the adsorption percentage.

III. Result and Discussion

The quality of the coconut shell adsorbent was assessed through a series of tests, including parameters such as moisture content, ash content, iodine adsorption capacity, and methylene blue adsorption capacity. These tests were conducted to determine whether the produced coconut shell adsorbent meets the standards specified in SNI No. 06-3730-1995.

In this study, coconut shell was used as the adsorbent, with an initial raw material weight of 2 kg. Following carbonization at 300°C, the yield of the coconut shell adsorbent was 64.03%. The adsorbent was activated using two methods: physical activation by heating in a furnace at 500°C for 1 hour, and physico-chemical activation by soaking the heated adsorbent in a 0.5 M H3PO4 solution. The purpose of activation is to increase the active sites on the adsorbent for more optimal adsorption.

Moisture content for physically activated coconut shell adsorbent was 5.06%, and for physico-chemically activated adsorbent, it was 4.9%. A lower moisture content indicates better adsorbent quality as excessive moisture can block the pores. The SNI 06-3730-1995 standard specifies a maximum moisture content of 15%, which both adsorbents met.

Ash content was 7.05% for physically activated adsorbent and 2.2% for physico-chemically activated adsorbent. Lower ash content is preferable as excessive ash can clog the pores, reducing the adsorbent's surface area. The maximum ash content according to SNI 06-3730-1995 is 10%, and both adsorbents were within this limit.

TABLE I
QUALITY OF COCONUT SHELL ADSORBENTS WITH SNI STANDARDS

Parameter	Physical Activation Adsorbent	Physical-Chemical Activation Adsorbent	SNI (1995)
Water Content	4,9 %	5,06 %	< 15 %
Ash Content	7,05 %	2,2 %	< 10 %
Absorbency towards iodine	761,58 mg/g	885,51 mg/g	> 750 mg/g
Absorbency towards Methylene Blue	302,946 mg/g	306,028 mg/g	> 120 mg/g

Iodine adsorption capacity was 761.58 mg/g for physically activated adsorbent and 885.51 mg/g for physico-chemically activated adsorbent. Higher iodine adsorption indicates better adsorption capacity. The minimum standard set by SNI 06-3730-1995 is 750 mg/g, which both adsorbents exceeded.

Methylene blue adsorption capacity was 305.946 mg/g for physically activated adsorbent and 306.028 mg/g for physico-chemically activated adsorbent. Higher methylene blue adsorption indicates a larger surface area of the adsorbent. The minimum standard according to SNI 06-3730-1995 is 120 mg/g, and both adsorbents surpassed this value.

A. The Influence of Adsorbent Mass and Contact Time on Adsorption towards Methylene Blue

The study was conducted with adsorbent mass variables of 5 grams, 10 grams, 15 grams, 20 grams, and 25 grams, and a contact time of 60 minutes to determine the optimal adsorbent mass for both physical activation and physical-chemical activation. Once the optimal adsorbent mass was identified, the contact time was varied at intervals of 15 minutes, 30 minutes, 45 minutes, 60 minutes, and 75 minutes. The analysis was performed using a UV-Vis spectrophotometer at a wavelength of 662 nm. The initial analysis of the methylene blue solution sample prior to adsorption revealed a concentration of 18.614 mg/L.

Based on the data presented in Table II, the initial concentration of the methylene blue solution was measured at 18.614 mg/L. For the physically activated adsorbent, the optimal adsorption performance was observed using a 10-gram sample, achieving a concentration of 0.859 mg/L. This indicates that the physically activated adsorbent effectively reduced the concentration of methylene blue in the solution.

In contrast, the physically-chemically activated adsorbent exhibited the best adsorption results with a 25-gram sample, resulting in a concentration of 0.765 mg/L. This higher mass of adsorbent led to a slightly lower concentration of methylene blue in the solution compared to the physically activated adsorbent.

TABLE IIII
EFFECT OF ADSORBENT MASS ON ADSORPTION TOWARDS METHYLENE BLUE SOLUTION FROM COCONUT SHELL ADSORBENTS WITH SNI STANDARDS

Adsorbent Mass (gr)	Contact Time (Minute)	Initial Concentration (mg/L)	Final Concentration (mg/L)	
			Physical Activated	Physical Chemical Activated
5		18,614	0,954	0,772
10		18,614	0,859	0,772
15	60	18,614	0,867	0,772
20		18,614	1,122	0,779
25		18,614	1,180	0,765

These findings suggest that both types of adsorbents were effective in removing methylene blue from the aqueous solution, with varying optimal conditions for each. The physically activated adsorbent showed higher efficiency with a smaller mass, whereas the physically-chemically activated adsorbent required a larger mass to achieve similar adsorption efficiency.

Based on the table above, the optimal contact time for the adsorption of methylene blue solution using the physical activation method was achieved with 10 grams of adsorbent and a contact time of 30 minutes. In contrast, the best results for the physical-chemical activation method were obtained with 25 grams of adsorbent and a contact time of 60 minutes.

TABLE IIII
EFFECT OF ADSORBENT CONTACT TIME TOWARDS ADSORPTION OF METHYLENE BLUE SOLUTION

Adsorbent Mass (gr)	Contact Time (Minute)	Initial Concentration (mg/L)	Final Concentration (mg/L)	
			Physical Activated	Physical Chemical Activated
5		18,614	0,954	0,772
10		18,614	0,859	0,772
15	60	18,614	0,867	0,772
20		18,614	1,122	0,779
25		18,614	1,180	0,765

The data indicate that the physical activation method reaches its highest adsorption efficiency with a relatively shorter contact time and a smaller adsorbent mass. This suggests that the surface area and active sites of the physically activated adsorbent are sufficient for optimal adsorption within a shorter duration.

Conversely, the physical-chemical activation method, which involves an additional chemical treatment, demonstrates improved adsorption efficiency with a higher adsorbent mass and longer contact time. This enhancement can be attributed to the increased number of active sites and the better-developed pore structure resulting from the chemical activation process. Consequently, the adsorbent requires more time to achieve maximum adsorption capacity.

These findings underscore the significance of optimizing both adsorbent mass and contact time to achieve efficient adsorption. They also highlight the differing dynamics between physical and physical-chemical activation methods in enhancing the adsorptive properties of the adsorbent material.

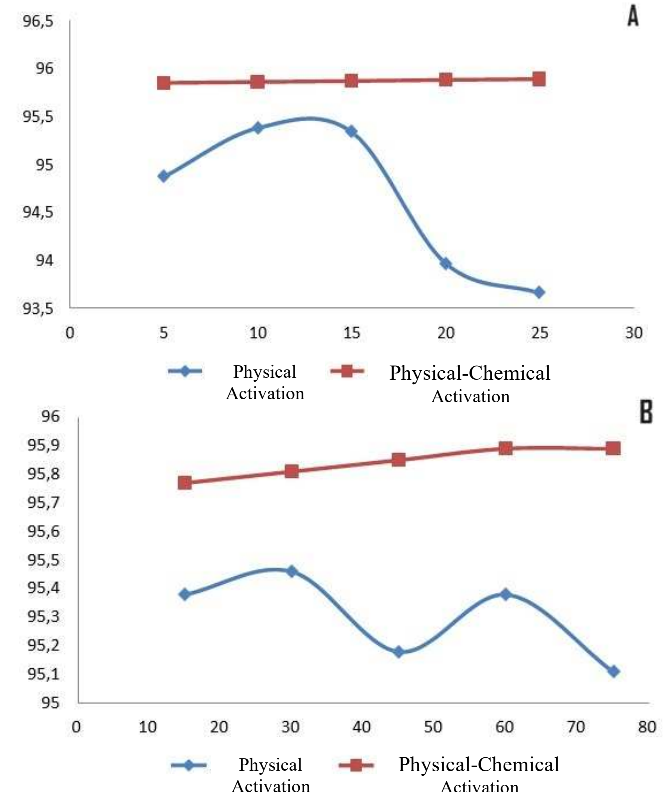

Fig. 3 Adsorption Graph of Physical Activation and Physical-Chemical Activation with Mass Variation (A) Adsorption Graph of Physical Activation and Physical-Chemical Activation with Contact Time Variation. (B)

The study investigates the influence of varying adsorbent masses (5 grams, 10 grams, 15 grams, 20 grams, 25 grams) and contact times (15 minutes, 30 minutes, 45 minutes, 60 minutes, and 75 minutes) on the adsorption efficiency of coconut shell-based adsorbents activated by physical and physical-chemical methods. The initial concentration of methylene blue solution before adsorption was measured at 18.614 mg/L using a UV-Vis spectrophotometer at a wavelength of 662 nm.

It was generally observed that an increase in the adsorbent mass resulted in a higher adsorption capacity and percentage. For physically activated adsorbents, a significant increase in adsorption percentage was noted from 5 grams to 10 grams, reaching a maximum adsorption efficiency of 95.38% at 10 grams. However, beyond this mass, the efficiency decreased due to the unutilized active sites on the adsorbent surface. In contrast, the physically-chemically activated adsorbents exhibited a continuous increase in adsorption efficiency, peaking at 95.89% with a 25-gram adsorbent mass. This improvement is attributed to the higher number of active sites available for interaction with the adsorbate.

Comparatively, the adsorption efficiencies achieved in this study are superior to those reported by previous researchers [10] reported a maximum adsorption efficiency of 92.43% with a 5-gram adsorbent mass, 93.745% with a 5-gram mass, and reported 80% efficiency with the same adsorbent mass.

Regarding contact time, the adsorption efficiency of physically-chemically activated adsorbents increased consistently from 15 to 75 minutes, achieving a maximum of 95.89% at both 60 and 75 minutes. Physically activated adsorbents showed a peak efficiency of 95.46% at 30 minutes, followed by a decline to 95.18% at 45 minutes, potentially due to operational anomalies such as pH variations or temperature fluctuations affecting the adsorption process. After 60 minutes, efficiency slightly increased to 95.38%, but a decline to 95.11% was observed at 75 minutes, indicating saturation.These findings surpass previous results [10] reported a maximum efficiency of 92.43% at 60 minutes, reported 93.745% at 15 minutes, and reported 80% at 60 minutes.

Both adsorbent mass and contact time significantly influence adsorption efficiency. Increasing the adsorbent mass enhances the number of active sites, improving adsorption until an optimal mass is surpassed, beyond which efficiency declines. Similarly, optimal contact time is crucial for maximum adsorption efficiency, as prolonged contact can lead to desorption and reduced efficiency. Optimal conditions for maximum adsorption are crucial and differ in various pattern depending on the adsorbent and operational parameters, aligning with theoretical expectations based on collision theory and adsorption kinetics.

IV. Conclusion

The coconut shell-based adsorbents, both physically and physically-chemically activated, were evaluated for their characteristics and adsorption efficiency in this study. The physically activated adsorbent exhibited a moisture content of 5.06%, ash content of 7.05%, and iodine adsorption capacity of 761.58 mg/g. Conversely, the physically-chemically activated adsorbent had a lower moisture content of 4.9%, ash content of 2.2%, and higher iodine adsorption capacity of 885.51 mg/g. These results confirm that the coconut shell-based adsorbents meet the standards outlined in SNI 06-3730-1995.

Optimal conditions for adsorption were determined as follows: for the physically activated adsorbent, the best performance was achieved using 10 grams of adsorbent with a contact time of 30 minutes, resulting in an adsorption percentage of 99.83%. Similarly, the physically-chemically activated adsorbent showed optimal performance with 25 grams of adsorbent and a contact time of 60 minutes, achieving an adsorption percentage of 99.87%.

Based on these findings, further research focusing on the contact time is recommended, as it plays a crucial role in the adsorption process of coconut shell-based adsorbents. This study underscores the potential of these adsorbents for water treatment applications, providing essential data for optimizing their use in environmental remediation efforts. Future investigations should explore additional factors affecting adsorption performance to enhance the efficiency and applicability of coconut shell-based adsorbents in practical settings.

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