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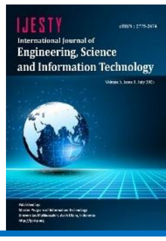


# Optimization Study Of Time And Mass Of Ketapang Fruit Shell Biosorbent in the Methylene Blue Batch Adsorption Process

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**Abstract**

Ketapang fruit shell (*Terminalia catappa*) has shown potential as a low-cost, eco-friendly biosorbent for removing synthetic dyes from aqueous solutions. This study aimed to determine the optimum conditions for contact time and adsorbent mass of ketapang fruit shell in the adsorption of methylene blue dye. The research involved several stages: preparation and chemical activation of the fruit shells, batch adsorption experiments, analysis of adsorption capacity and efficiency, and optimization using the Response Surface Methodology (RSM). Characterization of the adsorbent was conducted using UV-Visible spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, and Scanning Electron Microscopy (SEM) to analyze functional groups and surface morphology. The RSM approach was employed to evaluate the interaction effects of contact time and adsorbent mass, as well as to predict the optimal conditions for maximum dye removal. The optimum adsorption conditions were achieved at a contact time of 39.15 minutes and adsorbent mass of 0.416 g, resulting in an adsorption capacity of 1.935 mg/g and 100% removal efficiency. The regression model obtained from RSM was: $Y = 35.13437 + 334.35597A - 1.67299B + 0.088354AB - 402.98656A^2 + 0.018207B^2$**,** with a coefficient of determination $R^2 = 0.7857$ (78.57%)**,** indicating a good model fit. Furthermore, the adsorption behavior followed the Freundlich isotherm model, supported by an $R^2$ value of 0.787 (78.7%)**,** suggesting multilayer adsorption on a heterogeneous surface. These findings confirm the effectiveness of ketapang fruit shell as a promising adsorbent for methylene blue removal in wastewater treatment applications.

***Keywords***: *Adsorbent, Effective, Efficiency, Optimization, Solution.*

## 1. Introduction

Water is essential in every development aspect, related to all Sustainable Development Goals (SDGs), particularly for economic growth, healthy ecosystems, and life. Despite these numerous advantages, the textile industry often pollutes water with hazardous waste, threatening ecosystems and human health. Therefore, proper water management and controlling textile pollution are essential for global prosperity [1]. In 2021, the global textile dyes market was valued at USD 10.68 billion and is estimated to grow at 4.7% annually. The dye used on various fabrics contains chemicals that harm the environment. By 2023, dye production is expected to increase by more than 800,000 tons annually, mostly for the textile industry [2].

The textile and clothing industry accounts for approximately 12% of total national production in Indonesia, producing significant volumes of colored liquid waste [3]. Consequently, the government and environmental organizations have developed mitigation strategies to promote environmentally friendly waste processing. In textile waste, methylene blue is a common dye that can harm health and the environment [4] [5]. As a heterocyclic aromatic compound, when methylene blue enters the human body at high doses, it can accumulate and dimerize in cells. Although carcinogenic studies on mouse and rat cells have shown an increase in existing tumors [6] [7] [8].

Several technologies have been developed to remove pollutants from aqueous solutions, such as oxidation, ion exchange, membrane filtration, photocatalytic degradation, and adsorption. Among these technologies, adsorption is the main option due to its low cost but efficient performance. Some materials with high adsorption capacity, such as carbon nanotubes, nano-composites, and nanospheres, raise concerns regarding health potential. Using acrylic acid-methyl propane sulfonic acid (AMPS) to increase adsorption capacity may pose health risks. Although polymers are still commonly used, biosorption is preferred due to its environmentally friendly nature, including dead material biomass such as agricultural waste, with functional groups that bind dyes [9].

Studies around the world are searching for cheap and environmentally friendly adsorbents. One plant that attracts attention is ketapang tree (*Terminalia catappa Linn*). It is a multipurpose plant cultivated on roadsides, with the wood being used for construction and medicinal purposes. Generally, ketapang tree grows in coastal areas to a height of 25-40 meters and is widely distributed in Southeast Asia, India, Polynesia, Madagascar, Pakistan, Africa, South America, and Central America. Several investigations have explored the wood part of the plant as biodiesel [10] [11] , and protein sources [12]. Other functions include adsorbents [8, 13, 14] , pharmacy and beauty [15, 16], food ingredients [17] and briquettes [18]. Various studies have also proven that ketapang is a potential, cheap, and environmentally friendly plant, which does not interfere with food security. Therefore, this study aims to determine the optimum conditions for contact time and adsorbent mass of ketapang fruit shells.

## 2. Literature Review

### 2.1. Biosorption

Biosorption is an effective process for removing pollutants from water or other media by utilizing biological materials—such as bacteria, fungi, algae, or agricultural waste as biosorbents. This process encompasses both physical mechanisms (e.g., sorption and adsorption) and chemical mechanisms (e.g., ion exchange and complexation), operating independently of cellular metabolism [19]. It is particularly well-suited for the removal of heavy metals and other hazardous contaminants from industrial effluents and agricultural waste streams [20].

The primary advantages of biosorption include its low operational cost, high removal efficiency, and environmentally friendly nature . Moreover, the utilization of biological waste materials as biosorbents adds value to otherwise discarded biomass, thereby supporting sustainable waste management practices. However, biosorption performance is influenced by several operational parameters, such as pH, temperature, biosorbent dosage, and the type of biosorbent used [21].

In the present study, biosorption technology was employed to remove methylene blue (MB), a common thiazine-based cationic dye widely used in the textile industry. MB is known for its high solubility in water and persistence in the environment, making its removal from aqueous systems a significant environmental concern. Various biosorbents—including activated carbon derived from sugarcane bagasse, fungal biomass, and other plant-based materials—have been investigated for their effectiveness in MB adsorption [22]. The process of MB biosorption generally involves the binding of dye molecules to the surface of the biomass, driven by electrostatic interactions and surface complexation.

### 2.2. Methylene blue

Methylene blue (MB) is a synthetic heterocyclic aromatic compound with the molecular formula $C_{16}H_{18}ClN_3S$ and a molecular weight of 319.86 g/mol. It is highly soluble in water, with a solubility of $4.36 \times 10^4$ mg/L, and has a melting point of approximately 105°C [23]. Due to its vibrant color and stability in solution, MB is commonly used in textile dyeing, biological staining, and as a redox indicator in various laboratory procedures. However, these same properties also contribute to its persistence in aquatic environments when discharged as waste, posing a challenge for conventional water treatment methods.

Environmentally, the presence of methylene blue in water bodies can significantly reduce light penetration, thereby disrupting photosynthetic activity in aquatic ecosystems. Prolonged exposure can also cause adverse health effects in humans and animals, including irritation of the skin, disturbances in the digestive system, and in severe cases, cyanosis—a condition characterized by bluish discoloration of the skin due to lack of oxygen in the blood .As a result, methylene blue is considered a pollutant of concern and its removal from wastewater is critical for protecting both human health and ecological balance [24].

## 3. Methods

The pieces of ketapang fruit were collected from the parking area of the Faculty of Engineering, Malikussaleh University, Lhokseumawe, North Aceh, Aceh province, Indonesia. Other materials included methylene blue or MB (E. MERCK with high purity ≥ 89 %), Sodium hydroxide (Sodium hydroxide puriss. p.a., ACS reagent, reag. Ph. Eur., K ≤ 0.02%, ≥ 98%, pellets), and distilled water.

### 3.1. Preparation of Ketapang Fruit Shells

Ketapang fruit shells were cut into 1-2 cm sizes, washed, and dried in the sun until dry. The drying process was continued in an oven (UN110 Universal Oven) at $105^{O}$C until the water content was constant, followed by burning in a muffle furnace at $600^{O}$C for 4 hours. Ketapang fruit shells samples were stored in a desiccator (Duran Vacuum Desiccator 30cm) for 6 hours, followed by a comminution process with a particle size of 100 mesh. Subsequently, the powder form was subjected to preliminary testing for functional group identification using FTIR (FTIR Nicolet 8700 spectrometer (SHIMADZU) and SEM.

### 3.2. Ketapang Fruit Shells Adsorbent Activation

A total of 100 ml of Sodium hydroxide solution (0.1 M) was added to ketapang fruit shells powder and soaked for 24 hours at room temperature. After filtering, the residue was washed with distilled water to achieve a neutral pH. Ketapang fruit shells samples were dried in an oven at $105^{O}$C until the water content was constant.

### 3.3. Adsorption of Methylene Blue

A total of 50 ml of 50 mg/L methylene blue solution was added to the Erlenmeyer (Duran Erlenmeyer Flask 100 ml), followed by ketapang fruit shells powder with a mass of 0.1 each, 0.3, and 0.5 grams. The Erlenmeyer containing methylene blue solution and ketapang fruit shells were stirred in a stirrer (Orbital Shaker Laboratory) at a speed of 100 rpm at 30 operating times each, 45 and 60 minutes. The solution in the batch adsorption process was filtered. The filtrate obtained was analyzed for its content using a UV-Vis spectrophotometer with a wavelength of 663 nm, FTIR, and SEM. An analysis was carried out by checking the capacity and adsorption percentage, each using the formula (1) and (2):

$$q_e = \frac{V}{m} \times (C_o - C_e) \tag{1}$$

$$\%\ \text{adsorption} = \frac{(Co\text{-}Ce)}{Co} \times 100\% \tag{2}$$

Where Q, V, m, Co, Ce and respectively are adsorption capacity (mg/g), volume of solution (L), adsorbent weight (g), initial and final concentration (mg/L).

**Table 1.** Factors in the independent variable

| Independent variable | Alpha code and range | | |
|---|---|---|---|
| | -1 | 0 | +1 |
| Time | 30 | 45 | 60 |
| Adsorbent mass | 0,1 | 0,3 | 0,5 |

### 3.4. Design of Optimization Experiments Response Surface Method

Statistical experimental design is efficient for developing experiments to produce valid and objective conclusions after data analysis. The two major applications of experimental design are distilled to determine the factors influencing the experiment and optimal conditions. In this study, Design Expert 13.0 software (Stat-Ease Inc., Minneapolis, MN, USA) was applied for regression and graphical analysis of the data. The response surface method explored response patterns and determined the best combination of variables expected to produce optimal conditions. Table shows the experimental design in this study which included two variables, namely A (contact time) and B (adsorbent mass).

## 4. Results and Discussion

### 4.1. Adsorption Isotherms and Optimum Conditions

The linearization of the Langmuir and Freundlich equation tests differs, as shown in Figure 1. The Freunlich isotherm model describes the methylene blue batch adsorption process by ketapang fruit shells based on these linear values. Good linearization graph and a correlation value (R2) is close to 1. Using ketapang fruit shells as adsorbent the characterization of the methylene blue batch adsorption mechanism can be effectively carried out with the Langmuir adsorption isotherm model.[6] also obtained similar results using palm leaf frond adsorbent.

The relationship between Ce/Qe and Ce which is derived from the equation y = 0.0249x + 0.0527 shows the Langmuir isotherm pattern curve and is used to calculate the slope and intercept values to obtain the quantities of the constants KL, (the Langmuir isotherm constant (L/mg)) RL, and qmax. The Langmuir isotherm equation's calculation results yielded the constant values which included KL = 0.469 L/mg, correlation coefficient (R2) = 0.0527, RL= 0.0408 L/g, and qmax = 18.939 mg/g. The relationship pattern between Log Qe and Log Ce is created to produce the Freundlich isotherm, with the equation obtained y=-0.3501x+1.1188, which will be used for the slope and intercept of the graph to obtain the constant values Kf and 1/n. From the calculation results, the correlation coefficient (R2) = 0.7857, the slope value (1/n) = 2.853 and the intercept value (Kf) = 0.049 L/mg.

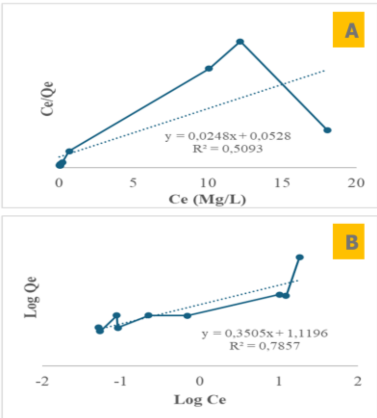


**Fig 1.** Isotherm curve: A. Langmuir; B. Freundlich

Based on the optimization results shown in Table 2 and Figure 2, the combination of independent variable levels that provides the optimal response value is a mass of 0.416 g and a contact time of 39.15 min. The desirability value obtained is 1, showing the program's ability to meet the optimization criteria set for the final product. This shows that as the desirability value becomes 1, program's ability increases to produce good products [6].

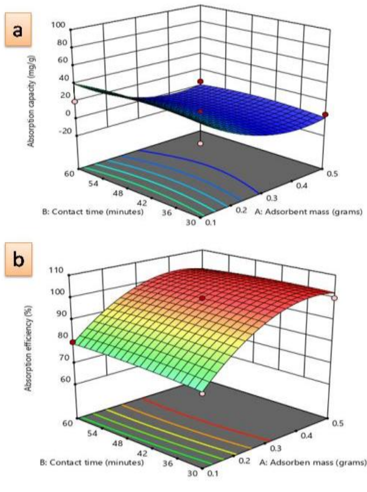


**Fig 2.** The plot of 3D surface response diagrams of contact time (min) and adsorbent mass (g) on adsorption capacity (A) and efficiency (B).

**Table 2.** Optimum conditions for methylene blue batch adsorption by ketapang fruit shells adsorbent

| Contact time (min) | Adsorbent mass (g) | Adsorption capacity (mg/g) | Percent Adsorption (%) | Desirability |
|---|---|---|---|---|
| 39.15 | 0.416 | 1.935 | 100 | 1 |

## 4.2. Optimization Model Validation

Validation of prediction results against experimental data is presented in Table 3, showing that the percentage of error at each point is significantly small. Therefore, the proposed model obtained in equation (3) is valid enough to express the relationship between the independent and the dependent variables. The results against experimental data are a method to prove the validity of the proposed model obtained in equation (3).

$$Y_1 = 35{,}13437 + 334{,}35597A - 1{,}67299B + 0{,}088354AB - 402{,}98656A^2 + 0{,}018207B^2 \qquad (3)$$

**Table 3.** Model validation

| Run | Independent variable | | Dependent variable | | Error |
|---|---|---|---|---|---|
| | A: Adsorbent mass (g) | B: Contact time (min) | Adsorption capacity (mg/g) | Prediction ($Y_1$) | (%) |
| 1 | 0.1 | 30 | 18.899 | 39.26 | 1.07 |
| 2 | 0.5 | 30 | 4.994 | 1.18 | 3.22 |
| 3 | 0.1 | 60 | 19.955 | 40.03 | 1 |
| 4 | 0.5 | 60 | 4.99 | 0.88 | 4.6 |
| 5 | 0.017 | 45 | 93.093 | 67.86 | 0.3 |
| 6 | 0.58 | 45 | 4.284 | 13.25 | 2 |
| 7 | 0.3 | 24 | 8.296 | 6.23 | 0.33 |
| 8 | 0.1 | 30 | 8.217 | 6.25 | 0.31 |
| 9 | 0.5 | 30 | 8.318 | 6.11 | 0.36 |

## 4.3. Analysis of Variance (ANOVA) Regression Model

The model can be declared to have a significant influence when the probability value <0.05. However, when the probability value (Prob > F) is greater than 0.1, the model is in significant. As shown in Table 4, ANOVA for the model and variable A has a probability value (Prob > F) greater than 0.1. This indicates that the quadratic model and variable A only slightly influence adsorption capacity. Meanwhile, variable A is still included in the model, considering the possibility of a significant influence on adsorption capacity.

**Table 4.** Analysis of variance in the total amount of adsorption

| Source | Sum of square | DF | Mean square | F value | Prob > NF | Description |
|---|---|---|---|---|---|---|
| Model | 5062.30 | 5 | 1012.46 | 4.17 | 0.0446 | Significant |
| A-Mass | 2982.39 | 1 | 2982.39 | 12.29 | 0.0099 | |
| B-Time | 0.1108 | 1 | 0.1108 | 0.0005 | 0.9835 | |
| AB | 0.2810 | 1 | 0.2810 | 0.0012 | 0.9738 | |
| $A^2$ | 1807.56 | 1 | 1807.56 | 7.45 | 0.0294 | |
| $B^2$ | 116.74 | 1 | 116.74 | 0.4811 | 0.5103 | |
| Residual | 1698.57 | 7 | 242.65 | | | |
| Lack of Fit | 1698.57 | 3 | 566.19 | | | |
| Pure error | 0.000 | 4 | 0.0000 | | | |
| Cor Total | 6760.88 | 12 | | | | |

The data in Table 5 presents a feasibility test of methylene blue batch adsorption capacity model by ketapang fruit shells adsorbent. Optimization analysis of the response surface method obtained a third-order model regression equation using a quadratic model. The model's accuracy can be observed from the R-squared ($R^2$) value of 0.8099. When $R^2$ value is close to 1, the model is better at predicting the response. In the sum of the square test, a model is declared appropriate when the difference between the Adj $R^2$ and Pred $R^2$ values is smaller than 0.2.

The result shows that the value of Adj $R^2$ is 0.6742, and Pred $R^2$ is -0.3516. This shows that this model is unsuitable because the difference between the values of Adj $R^2$ and Pred $R^2$ is more significant than 0.2. A model is also considered to be good when the ratio of Adeq Precision is more than 4. As shown in Table 5, the ratio of Adeq Precision is 6.2002, which is sufficient for this model to be used.

**Table 5.** Quadratic model data on adsorption capacity

| Source | Sum of squares | DF | Mean square | F value | Prob> F | Description |
|---|---|---|---|---|---|---|
| **Sequential Model Sum of Squares** | | | | | | |
| Mean vs Total | 3211.49 | 1 | 3211.49 | | | |
| Linear vs Mean | 2982.5 | 2 | 1491.25 | 3.95 | 0.0545 | |
| 2FI vs Linear | 0.2810 | 1 | 0.2810 | 0.0007 | 0.9799 | |
| Quadratic vs 2FI | 2079.52 | 2 | 1039.76 | 4.28 | 0.0509 | Suggested |
| Cubic vs Quadratic | 1169.63 | 2 | 584.81 | 5.53 | 0.0541 | Aliased |
| Residual | 528.95 | 5 | 105.79 | | | |
| Total | 9972.37 | 13 | 767.11 | | | |
| **Model Summary Statistics** | | | | | | |
| **Source** | **Std Dev** | **$R^2$** | **Adj $R^2$** | **Pred $R^2$** | **PRESS** | **Description** |
| Linear | 19.44 | 0.4411 | 0.3294 | -0.1917 | 8057.25 | |

| | | | | | | |
|---|---|---|---|---|---|---|
| 2FI | 20.49 | 0.4412 | 0.2549 | -0.5086 | 10199.23 | |
| Quadratic | 15.58 | 0.7488 | 0.5693 | -0.7866 | 12078.74 | Suggested |
| Cubic | 10.29 | 0.9218 | 0.8122 | -4.0071 | 33852.57 | Aliased |

## 4.4. Characteristics of Ketapang Fruit Shells Biosorbent

Ketapang fruit shells have been explored in various studies as an effective adsorbent for overcoming environmental pollution problems. As presented in Figure 3, ketapang fruit shells are a multipurpose plant widely cultivated on roadsides, with the wood being used for house construction and medicinal materials. Although it grows naturally in coastal areas, the plant is often found in tropical areas to a height of 25-40 meters. In this study, visualization of methylene blue batch adsorption results by CBK biosorbent showed significant color changes due to the effectiveness of biosorbent performance based on time variables of 30, 45, and 60 min.

Figure 4 shows the FTIR spectrum of the biosorbent before and after methylene blue batch adsorption. The wave numbers of several functional groups in ketapang fruit shells shift due to chemical interactions between the biosorbent and methylene blue. Strong O-H stretching is observed at 3200–3600 $cm^{-1}$, while the peak at 2850–3000 $cm^{-1}$ is associated with C-H stretching. C=O is observed at 1820–1600 $cm^{-1}$, related to aromatic C=C. The characteristics of IR adsorption before and after the adsorption of methylene blue batches on ketapang fruit shells show that the presence of tannins and phenolic compounds, which have O-H functional groups, can interact to absorb methylene blue [23]. The presence of chemical interactions between the biosorbent and the dye was shown by a slight peak shift observed but the spectra were similar.


**Fig 3.** Parts of ketapang tree and visualization of MB batch adsorption results by ketapang fruit shells biosorbent with variable optimization of contact time and adsorbent mass.

In UV-Vis spectrophotometry, auxochrome groups including O-H, C-H, and C=O can increase the wave adsorption , raising the efficiency value. A subset of functional groups such as C-N is the chromophore group that affects the adsorption wavelength. The chromophore group can increase the wave adsorption capacity, thereby improving the efficiency when observed in a compound [25]. Due to the wider opening of ketapang fruit shells pores, the FTIR spectrum shows that the waves become sharper after activation with NaOH. This is caused by ionic bonds formed between NaOH and the surface of ketapang fruit shells adsorbent, which can increase the surface area of the adsorbent [26].

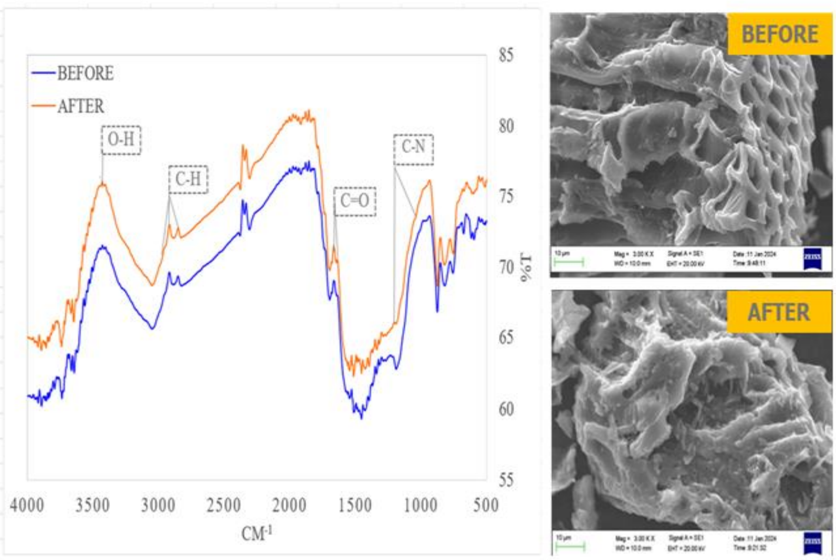


**Fig 4.** Infrared and visual SEM spectra before and after adsorption of MB batches by ketapang fruit shells biosorbent

The SEM analysis results of ketapang fruit shells show their surface morphology after the adsorption process. The surfaces and pores are observed to be covered by the methylene blue dye used in the adsorption process. Therefore, the adsorption capacity will decrease with more methylene blue being adsorbed onto the active surface. This is because the volume of the adsorbent pores will limit the number of adsorbate molecules that enter the adsorbent pores. When the adsorbent pores are filled with adsorbate molecules, saturation will occur causing a reduction in reactivity, and adsorption equilibrium is reached [27].

## 5. Conclusion

In conclusion, this study successfully optimizes the mass and contact time of ketapang fruit shells adsorbent for methylene blue batch adsorption. The results showed that the optimum conditions were obtained at 39.15 min and a mass of 0.416 g with an adsorption capacity of 1.935 mg/g and an efficiency of 100%. Furthermore, the model obtained was $Y = 35.13437 + 334.35597A - 1.67299B + 0.088354AB - 402.98656A^2 + 0.018207B^2$, with a correlation level of $R^2 = 0.7857$ (78.57%). The methylene blue adsorption process from ketapang fruit shells adsorbent was followed by the Freundlich isotherm model, based on the test results of the squared coefficient of determination $R^2$ 0.787 (78.757%)..

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