

REMOVAL OF NAPHTHOL DYE FROM BATIK WASTEWATER USING CHITOSAN FROM BLACK SOLDIER FLY PUPAL EXUVIAE

Putri Rayhani Rahmania, Arseto Yekti Bagastyo*

Department of Environmental Engineering, Institut Teknologi Sepuluh Nopember, Indonesia

Correspondence Author: bagastyo@enviro.its.ac.id

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ABSTRAK

Industri batik merupakan salah satu industri yang limbahnya berpotensi menjadi sumber pencemaran air karena limbah cair yang dihasilkan mengandung polutan seperti warna dalam konsentrasi tinggi. Untuk menyisihkan polutan yang terdapat dalam limbah, metode adsorpsi digunakan sebagai upaya agar limbah tidak mencemari sumber air. Penelitian ini membahas tentang penerapan kitosan dari eksuvia pupa black soldier fly (BSF) sebagai adsorben dalam adsorpsi zat warna naftol pada air limbah batik. Proses adsorpsi dilakukan menggunakan metode batch dengan tujuan untuk menganalisis efisiensi penyisihan zat warna dan kapasitas adsorpsi yang dipengaruhi oleh pH larutan dan dosis adsorben, serta menentukan model kinetika adsorpsi yang sesuai untuk diterapkan. Perubahan konsentrasi zat warna dianalisis serapannya menggunakan spektrofotometer. Hasil penelitian proses adsorpsi menunjukkan pH dan dosis adsorben optimal berada pada pH 4 dan 0,4 g/L dengan efisiensi penyisihan mencapai 92%. Efisiensi penghilangan menurun dengan meningkatnya pH. Semakin banyak jumlah adsorben maka efisiensi penyisihan semakin menurun. Namun setelah mencapai dosis optimal, penambahan massa kitosan tidak lagi memberikan peningkatan efisiensi penyisihan yang signifikan, hal ini menunjukkan bahwa titik jenuh telah tercapai. Proses adsorpsi cenderung mengikuti model kinetika orde dua semu dengan kapasitas adsorpsi maksimum sebesar 344,82 mg/g.

Kata kunci: adsorpsi, black soldier fly, kitosan, pewarna naftol.

ABSTRACT

The batik industry is one of the industries whose waste has the potential to become a source of water pollution because the wastewater produced contains pollutants such as dyes in high concentrations. To remove pollutants contained in the wastewater, the adsorption method is used as an effort to prevent the effluent from polluting water sources. This research discussed about the application of chitosan from black soldier fly (BSF) pupal exuviae as an adsorbent in the adsorption of naphthol dyes in batik wastewater. The adsorption process was carried out in batch process with the aim of analyzing the dye removal efficiency and adsorption capacity as affected by solution pH and adsorbent dosage, as well as determining the appropriate adsorption kinetics model to be applied. Changes in dye concentration were analyzed for absorbance using a spectrophotometer. The results of the adsorption process research showed that the optimal pH and adsorbent dose were at pH 4 and 0.4 g/L with removal efficiency reaching 92%. The removal efficiency decreased with increasing pH. The increasing amount of adsorbent, the removal efficiency decreased. However, after reaching the optimal dose, the addition of chitosan

mass no longer gave a significant increase in the removal efficiency, indicating that the saturation point had been reached. The adsorption process tends to follow a pseudo second-order kinetics model with a maximum adsorption capacity of 344.82 mg/g.

Keywords: adsorption, black soldier fly, chitosan, naphthol dyes.

INTRODUCTION

Batik industry is one of the important sectors in Indonesia's creative economy that has a major contribution to the local economy and culture (Rini Afriantari et al., 2020). However, the batik production process often produces a high volume of wastewater. About 80% of the amount of water used for batik is discharged as wastewater (Suharto et al., 2016). Batik production centers in one village area have the potential to produce high liquid waste with a volume of $\pm 568 \text{ m}^3$ per day with a production of around 1400 pieces of batik cloth per day (Riyanto et al., 2019). Liquid waste generated from batik production often contains a variety of harmful chemical compounds, including synthetic dyes such as naphthol. Naphthol ($\text{C}_{10}\text{H}_8\text{O}$) is an azo dye that is widely used because of its ability to produce bright and durable colors on batik cloth. This dye is an organic compound that cannot be biodegraded, making it difficult to decompose in nature (Mutiarra et al., 2022). In batik coloring, the dye is only used about 45%, while the remaining 55% will be disposed of as liquid waste. The presence of naphthol in wastewater poses a serious environmental problem due to its toxic nature and difficulty to be decomposed by natural processes (Rahmayanti et al., 2020).

Various waste treatment methods have been developed to reduce the negative impact of batik waste on the environment, one of which is the adsorption method (Abu Bakar et al., 2019; Riduan et al., 2022). Adsorption is the process of absorbing solutes from a solution onto the surface of a solid, which in this context is the adsorbent. Adsorption has proven to be one of the most effective and efficient methods in removing various types of colorants from wastewater. The advantages of this method include low operating costs, ease of implementation, and its ability to remove high concentrations of contaminants (Dutta et al., 2021). One of the widely used natural adsorbents studied in dye effluent treatment is chitosan. Chitosan is obtained from the deacetylation of chitin, which is a major component in the shells of crustaceans such as shrimps and crabs. Chitin is a long-chain polymer of N-acetylglucosamine, and chitosan is derived from chitin by removing the acetyl groups from the N-acetylglucosamine units. As a

biodegradable and biocompatible biopolymer, chitosan has various physicochemical properties that make it highly effective in adsorbing dyes. The structure of chitosan rich in amino and hydroxyl groups enables strong interaction with dye molecules through hydrogen bonding and electrostatic interactions (Nechita, 2017).

The increasing demand for chitosan has prompted the search for more sustainable alternative sources. One potential source is the pupal exuvia from Black Soldier Fly (BSF), which is a waste from the cultivation of these insects (Khayrova et al., 2019; Bakker, 2020). BSF is known to be an effective insect in recycling organic waste into useful biomass. This study aims to explore the potential of chitosan extracted from BSF exuvia as an adsorbent to remove naphthol dyes in batik waste. The use of BSF exuvia as a source of chitosan not only helps in the treatment of batik waste, but also provides added value to insect waste that is usually not utilized. Thus, this research contributes to an environmentally friendly and economical waste treatment technology, as well as supports sustainability and waste management efforts.

METHOD

Tools and Materials

The materials used in this study are pupal exuviae of black soldier fly, distilled water, HCl, HCOOH, HCl, NaOH, and naphthol dyes. The tools used in this research are magnetic stirrer and plate magnetic stirrer, tray, blender, sieve, oven, stopwatch, digital balance, laboratory glassware, visible spectrophotometer (Genesys 30), and cuvette.

Procedure

Preparation of Chitosan Adsorbent

The chitosan adsorbent used was obtained from the extraction of black soldier fly (BSF). Pupal exuvia of BSF were washed using water until clean, then dried in a drying oven at 105°C until the weight was constant. After drying, the sample was crushed into small particle powder using a blender. The crushed samples were then extracted with three main processes: demineralization, deproteination, and deacetylation. The extraction method used was based on research conducted by Triunfo et al., (2022). Demineralization was carried out by mixing the samples in 0.5 M formic acid (HCOOH) solution at a ratio of 1:10 (m/v) and stirring for 2 hours at room temperature. The demineralized material was then filtered, washed until neutral, and dried at 60°C overnight in an oven. Deproteination was carried out by adding 2 M NaOH to the

demineralized sample in a ratio of 1:10 (m/v), stirring for 2 hours at 80°C. The deproteinated material was filtered, washed until neutral, and dried at 60°C overnight in an oven. Afterward, deacetylation to obtain chitosan was carried out by adding 19 M NaOH solution at a ratio of 1:30 (m/v) and under constant stirring for 2 hours at 100°C. The residue was filtered, neutralized, and oven-dried at 60°C overnight.

Preparation of Naphthol Standard Solution and Calibration Curve

Naphthol 1000 mg/L standard solution was made by dissolving a mixture of naphthol dye powder and azo salt as much as 0,25 g with distilled water in a 250 mL volumetric flask. Furthermore, other working solutions were made with concentrations of 10; 20; 30; 40; 50; 60; 70; 80; 90; 100; 200; 400; 600; and 800 mg/L by diluting the naphthol solution. To analyze the dye concentration in batik waste, first determine the wavelength of naphthol using a spectrophotometer. Measurements were made with an initial naphthol concentration of 100 mg/L until the wavelength at maximum absorption was obtained. Naphthol solutions with several concentrations previously made were measured for absorbance using a spectrophotometer at the optimum wavelength obtained. Furthermore, the results obtained can be plotted the absorbance value against the concentration so that a naphthol standard solution calibration curve is obtained. Determination of naphthol dye concentration was obtained by plotting the absorbance value obtained from the observation into a curve.

Adsorption Process

Adsorption was carried out by contacting 100 mL of artificial waste containing naphthol dye and adsorbent in an erlenmeyer. The process was carried out in batch mode using a magnetic stirrer at 250 rpm for 1 hour. The adsorbent used was chitosan extracted from BSF as much as 0.02; 0.04; 0.06; 0.08; and 0.1 gram. After the adsorption process is complete, the sample is filtered to separate the adsorbent from the solution. Experiments to determine the kinetics of adsorption were carried out by contacting waste and chitosan adsorbent at the optimal pH and dosage. Sampling was carried out every 15 minutes for 1.5 hours. With a spectrophotometer, the absorbance was observed at the wavelength of maximum absorption to determine the dye concentration. The percentage of naphthol dye removal (%R) adsorbed by chitosan adsorbent was calculated using equation (1). The adsorption capacity, q was calculated using equation (2).

$$\%R = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$q \text{ (mg/g)} = \frac{(C_0 - C_e) \times V}{W} \quad (2)$$

where C_0 and C_e (mg/L) are the initial and final concentrations of naphthol dye solution, q (mg/g) is the amount of dye adsorbed by the adsorbent, V (L) is the initial volume of dye solution, and W (g) is the weight of the adsorbent.

RESULT AND DISCUSSION

Determination of Naphtol Dyes Standard Curves

The maximum wavelength was first determined using naphthol dye solution in the wavelength range of 400 - 700 nm. Based on spectrophotometer measurements in this study, the highest absorbance was produced at a wavelength of 600 nm. The wavelength of 600 nm was then used for the determination of naphthol dye concentration in the adsorption process.

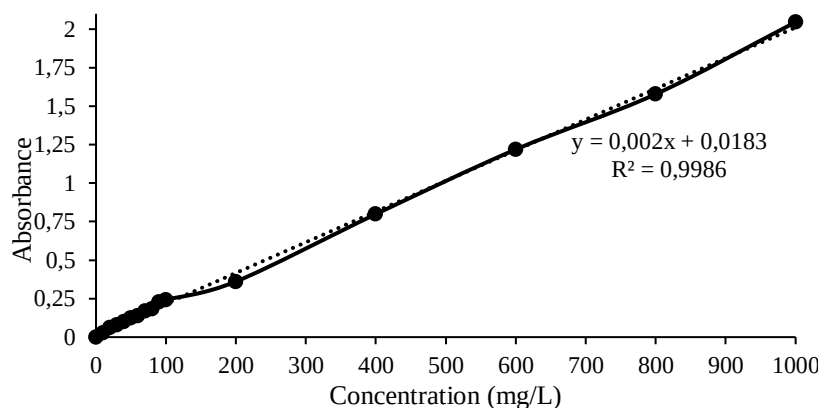


Figure 1. Calibration curve of naphthol dyes

The determination of the standard calibration curve aims to determine the absorbance measurement results of naphthol dyes before and after the adsorption process. The regression equation generated can be used to determine the concentration of the sample solution with the provisions of the y-axis is the absorbance while the x-axis is the concentration of naphthol dye. Figure 1. shows the correlation coefficient (R^2) value obtained is close to 1, indicating that the regression equation is linear and the effect of concentration on absorbance is 99.8%.

Effect of pH on Adsorption of Naphthol Dyes by Chitosan from Black Soldier Fly

Adsorption test was conducted to analyze the ability of BSF chitosan to adsorb naphthol dye contained in batik waste. Many factors affect the adsorption process, such as pH, adsorbent dosage, and contact time. Variations in the initial pH of the naphthol dye solution and adsorbent

dosage were carried out to determine the optimum conditions for naphthol dye adsorption using chitosan adsorbent.

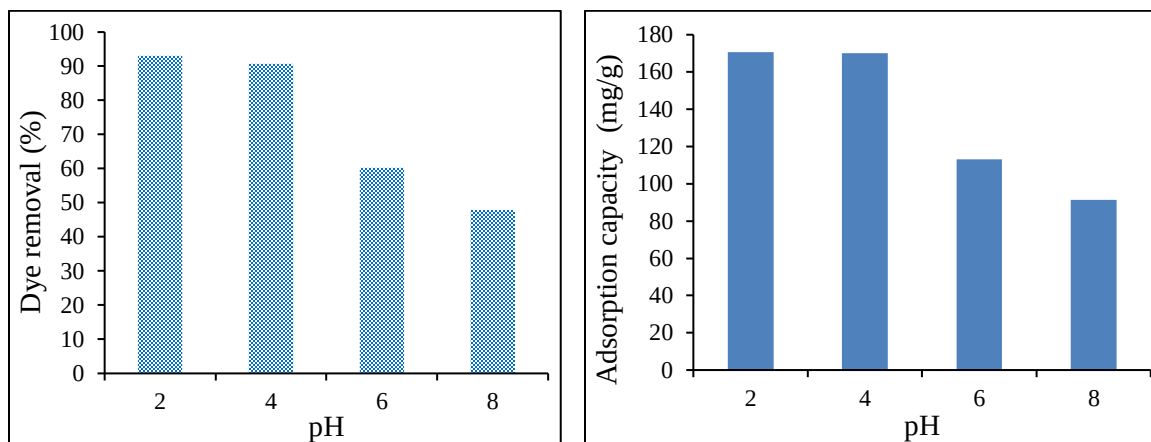


Figure 2. Effect of pH on dye removal and adsorption capacity by chitosan adsorbent

The effect of the initial pH of the solution on the adsorption of naphthol dye with chitosan is shown in Figure 2. In this study, the pH value in the effluent was adjusted using HCl solution. The results showed that the initial pH of the solution had a significant effect on the efficiency and adsorption capacity of chitosan in removing naphthol dye. The highest efficiency was achieved at pH 2 with 92% dye removal and 170.6 mg/g adsorption capacity. Chitosan successfully reduced naphthol dye from a concentration of 150 mg/L to around 10 mg/L. Significant color reduction also occurred at pH 4, with 90% dye removal. The color removal efficiency decreased as the pH of the solution increased further. At pH 6 and 8, the adsorption efficiency dropped to about 60% and 47%, respectively. At the same time, the adsorption capacity also decreased as the pH increased. This relationship indicates that the adsorption capacity and percentage removal of dye are directly related at lower pH conditions. The increase in removal efficiency at acidic pH is due to the protonation of amine groups on chitosan which enhances the electrostatic interaction with naphthol molecules. At low pH, the amine group ($-NH_2$) on chitosan is protonated to $-NH_3^+$, which results in a positively charged adsorbent surface (Safitri & Rahmayanti, 2020). Naphthol dyes generally contain sulfonate groups ($-SO_3^-$) which are negatively charged (Rahmayanti et al., 2021). The electrostatic interaction between the positively charged chitosan surface and the negatively charged naphthol dye promotes the adsorption process, so the adsorption efficiency increases at low pH. At higher or alkaline pH, there will be

a decrease in efficiency due to the deprotonation process of amine groups, which has a significant impact on electrostatic interactions in the system. As the pH increases, the initially protonated amine group deprotonates to a neutral form. This process reduces the positive charge on the molecule, which in turn weakens the electrostatic interaction with negatively charged species in the solution. As a result, the adsorption strength between the target molecules and the adsorbent surface decreases (Mishima et al., 2017). Similar results were obtained by Naghizadeh et al., (2017) which reported the adsorption efficiency of reactive blue dye was higher under acidic conditions.

Effect of Adsorbent Dosage on Adsorption of Naphthol Dyes by Chitosan from Black Soldier Fly

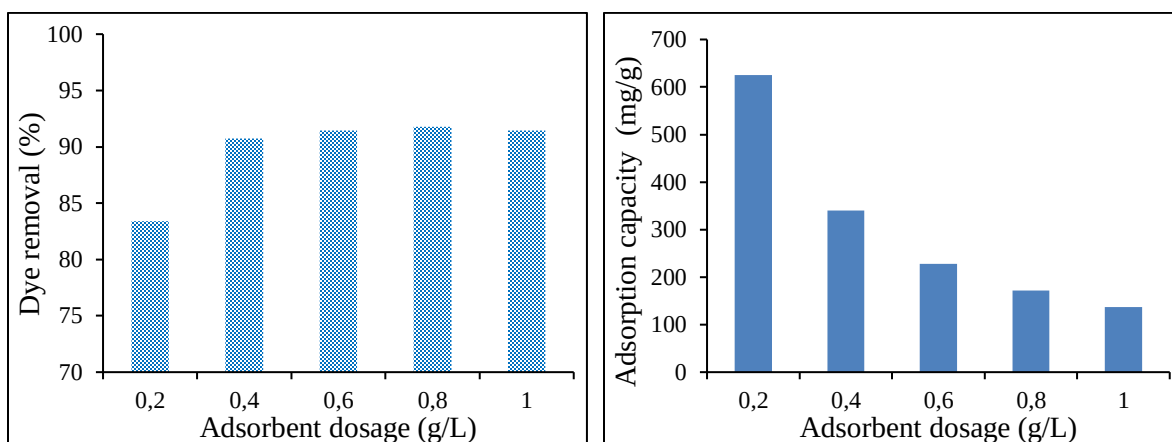


Figure 3. Effect of adsorbent dosage on dye removal and adsorption capacity by chitosan adsorbent

Determination of the optimum adsorbent dosage is very important in designing an efficient adsorption system. An adsorbent dose that is too low can result in inadequate dye reduction efficiency, while an adsorbent dose that is too high can lead to excessive and uneconomical adsorbent usage. Figure 3. shows the relationship between adsorbent dosage with removal efficiency and adsorption capacity of naphthol dye. It was found that the removal efficiency of naphthol color by chitosan increased with the increase of adsorbent dosage. The highest removal efficiency was achieved at 0.8 g/L adsorbent dosage with 91.76% removal. In contrast, the adsorption capacity of naphthol dye by chitosan decreased as the adsorbent dosage increased. The highest adsorption capacity was achieved at 0.2 g/L adsorbent dosage with a value of 625 mg/g. Based on the results obtained, the adsorption process was optimum at 0.2 g/L adsorbent dosage

due to its high removal efficiency and adsorption capacity. Increasing the adsorbent dosage causes an increase in naphthol dye removal efficiency due to the availability of more active sites to bind dye molecules. At higher adsorbent dosages, the surface area and number of active sites available for adsorption increase. This allows more dye molecules to interact with the adsorbent and be adsorbed from the solution. However, after reaching the saturation point, a further increase in adsorbent dosage does not provide a significant increase in removal efficiency due to the limited number of dye molecules remaining in solution. This indicates that the adsorption system has reached equilibrium, where most of the active sites have been occupied by dye molecules (Aljeboree et al., 2017). On the other hand, an increase in adsorbent dosage results in a decrease in adsorption capacity. At higher adsorbent dosages, the ratio of dye to active sites becomes lower, so not all active sites can be utilized efficiently. This reduces the accessibility of the dye to these sites resulting in a decrease in adsorption capacity per unit mass of adsorbent (Kamranifar & Naghizadeh, 2017).

Determination of Adsorption Contact Time of Naphthol Dyes by Chitosan from Black Soldier Fly

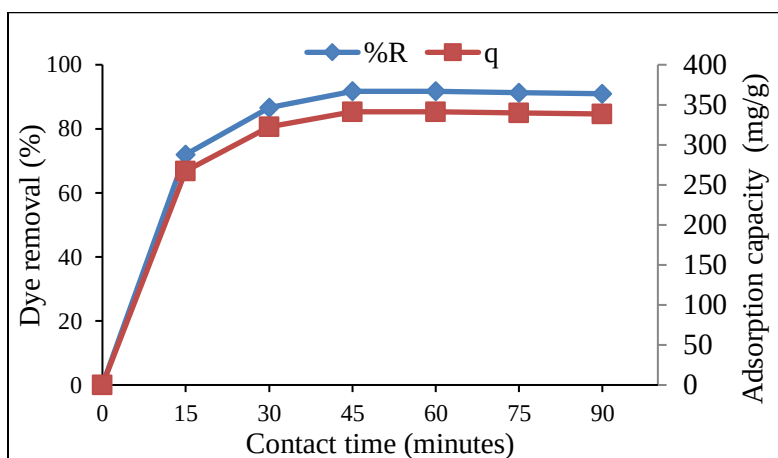


Figure 4. Determination of contact time on dye removal and adsorption capacity by chitosan adsorbent

Based on the optimum conditions of pH and adsorbent dosage obtained, the effect of contact time on the adsorption capacity and removal of naphthol dye by chitosan adsorbent was evaluated. The results in Figure 4 show that an increase in contact time results in an increase in adsorption capacity and percentage removal of dye until it reaches equilibrium. The percentage of dye removal (%R) and adsorption capacity (q) reached maximum values of about 91.7% and 341

mg/g at 45 min contact time, respectively. After this point, increasing the contact time gave a decrease in the adsorption capacity and percentage removal of the dye. At the beginning of the adsorption process, the adsorption capacity and percentage removal of dye increased significantly with increasing contact time. This can be attributed to the presence of numerous active sites on the surface of the chitosan adsorbent, which remain unoccupied during the initial stage. The dye molecules quickly diffuse from the solution to the adsorbent surface and attach to the available active sites. However, as the contact time increases, the rate of increase in adsorption capacity and percentage removal of dye gradually slows down until it finally reaches an equilibrium state. Most of the active sites have been occupied by dye molecules at an early stage, so the number of vacant sites available for further adsorption is limited. As a result, the rate of adsorption and dye removal slows down and eventually reaches equilibrium (Moosa et al., 2016).

Adsorption Kinetics Studies

Analysis of adsorption kinetics aims to understand the mechanism and rate of adsorption, thus determining the most appropriate kinetic model to describe the adsorption process. Adsorption kinetics are evaluated using two commonly used models, namely the pseudo-first order kinetics model and the pseudo-second order kinetics model. The kinetic models are used to obtain rate constants and equilibrium adsorption capacities. The solution adsorption rate and adsorbate residence time at the solution-adsorbent interface can be calculated using these models. The linear equations of pseudo first-order (3) and pseudo second order (4) are:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (4)$$

The values of k_1 of the pseudo first-order model and q_e were calculated from the slope and intercept of the plot of t versus $\ln(q_e - q_t)$ (Figure 5a). In contrast, k_2 of the pseudo second-order model and q_e were calculated from the slope and intercept of the plot of t versus t/q_t (Figure 5b). The experimentally calculated parameter values of the reaction order kinetics are summarized in Table 1.

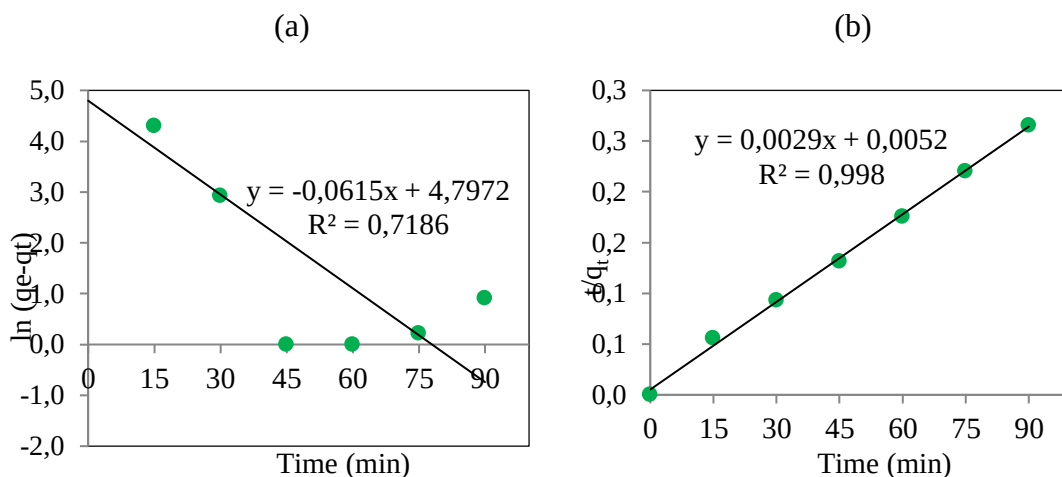


Figure 5. Pseudo first-order adsorption kinetics model (a), Pseudo-second-order adsorption kinetics model (b)

Table 1. Adsorption kinetics parameters

Adsorption Kinetics	Kinetics Parameters
Pseudo-frst order	$k_1 = 0,0615 \text{ min}^{-1}$ $q_e = 121,17 \text{ mg/g}$ $R^2 = 0,718$
Pseudo-second order	$k_2 = 0,0016 \text{ min}^{-1}$ $q_e = 344,82 \text{ mg/g}$ $R^2 = 0,998$

The value of the correlation coefficient (R^2) and reaction rate constant (k_2) in the second-order kinetic model is higher than the first-order kinetic model. Based on the calculation of pseudo second order kinetics, the calculated value of adsorption capacity at equilibrium condition (q_e) is 344.82 mg/g. This result is close to the experimental q_e value of 341.25 mg/g. Therefore, the adsorption process on chitosan adsorbent to adsorb naphthol dye shows a fit with the second-order kinetic model. The pseudo second-order kinetic model assumes that the adsorption capacity is proportional to the square of the number of active sites occupied on the adsorbent surface. The fit of naphthol dye adsorption data by chitosan to the pseudo second-order pseudo model indicates that the interaction between dye molecules and active sites on the chitosan surface is dominated by chemisorption. Chemical interactions between functional

groups on the chitosan surface, such as amine and hydroxyl groups, and Naphthol dye molecules contribute to the high adsorption efficiency (Wang et al., 2017).

CONCLUSION

The experimental findings indicate that chitosan derived from black soldier fly pupal exuviae is a promising adsorbent for removing naphthol dyes from batik wastewater. The dye removal efficiency was significantly affected by the initial pH and adsorbent dosage used. The removal efficiency improved with a higher dose of the adsorbent and lower pH levels. The adsorption process carried out under optimum conditions of pH 4 with an adsorbent dose of 0.2 g/L for 45 min showed a removal of naphthol dye of 91.7%. The experimental data showed an adsorption capacity of 341 mg/g and fit the pseudo second order kinetics model. The results of this study have great potential to be applied on a larger industrial scale, considering that chitosan is an environmentally friendly and easily available material. This method can be adapted to address other types of batik waste containing different dyes, with adjustments to operational conditions such as pH and adsorbent concentration. Implementation on a wider scale can help the batik industry meet environmental standards and reduce negative impacts on aquatic ecosystems.

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