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Performance of Activated Carbon from Date Seeds for Methylene Blue Treatment

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Abstract

Products that employ methylene blue dyes in their manufacturing processes leak harmful substances into the environment as waste. Activated carbon is used as an adsorbent to treat methylene blue because of a unique characteristic that makes it capable of being an excellent adsorbent in the wastewater treatment process. This research aims to develop an activated carbon using waste date seeds to treat methylene blue. It employed a chemical activation method to produce date seeds-activated carbon. Date seed samples have been through an impregnation ratio which 3:1 by using KOH solution for 2 hours. Then, the activation process was carried out in a furnace at 900 °C for 1 hour at a heating rate of 15 °C min^{-1}. The date seeds sample must be thoroughly processed to remove all excess chloride from KOH by using HCl and distilled water. Since increasing the amount of DSAC utilized decreases color more efficiently, it can be employed as an effective absorbent. The date seed was characterized using FTIR to identify functional groups and chemical bonds involved and SEM analysis to identify its morphology of activated carbon. Based on COD, turbidity, and color analysis, the value obtained increased proportionally with dosages 10 g, 20 g, 30 g, and 40 g. DSAC adoption showed positive results for color removal but not for COD and turbidity. 40 g of DSAC achieves an excellent result, removing 96.61% of the color from methylene blue. As the dosage of DSAC used increased, a greater reduction of color was achieved.

1. Introduction

Textile effluents are major sources of water contamination because they are chemically rich and contain large amounts of complicated chemical components. Textile companies generate a greater volume of effluent during the wet process. The wastewater generated from these industries has a high chemical oxygen demand (COD), pH, color, turbidity, biochemical oxygen demand (BOD), and total solids.

Dyes are colorless chemical compounds that can be used to color other substances. The dyes, mainly in the form of aromatic and heterocyclic compounds used in these industries to meet the color requirement, are highly toxic to the environment and mutagenic [1]. Highly colored synthetic dyes will increase water turbidity and prevent sunlight from penetrating the water. Methylene blue dye is a major contributor to waste discharged water.

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Methylene blue is widely used in a variety of industries, including silk, wood, and linen, and may leak many such materials into the environment as waste. It is a cationic dye with high resistance to light and heat. Around 10%-15% of the dyes are lost in the effluent during the dyeing process, and it might create a considerable quantity of COD and BOD in the wastewater [2]. Adsorption is one of the most cost-effective wastewater treatment techniques. It is an interfacial process in which gaseous or solute components gather on the surface of adsorbent materials [3]. This phenomenon is related to Van-der-Waals forces, which are physically attractive forces that bind gaseous and solute molecules.

Activated carbon is commonly used for the treatment of wastewater in the textile industry. Many companies utilize activated carbon for purifying effluents because it is a low-cost raw material source. This is because organic materials with a high lignocellulosic content are often where activated carbon may be found. This study produces activated carbon, which is found in date seeds. Date seeds are classified as dry waste because they are lightweight and large, and they accumulate in large quantities, posing a serious problem because they are one of the environmental pollutants when burned, in addition to providing a home for rodents and insects. Due to their good natural structure, lignocellulosic composition, and low ash content, date seeds are suited for the preparation of activated carbon. They are made up of 42% cellulose, 18% hemicellulose, 25% sugar, and other chemicals, 11% lignin, and 4% ash [4]. Thus, this research aims to evaluate the performance of activated carbon from date seeds for methylene blue treatment.

2. Materials and Methods

2.1 Materials

Date seeds were collected and chemical solutions such as Methylene Blue, Potassium Hydroxide (KOH), and Hydrochloric Acid (HCl) were prepared in a 1000 ml volumetric flask. The methylene blue was dissolved with distilled water to produce a 100 mg/L methylene blue solution. The potassium hydroxide (KOH) and hydrochloric acid were prepared to produce 0.1 M for each chemical. These chemicals were used in the preparation of DSAC.

2.2 Preparation of DSAC

Preparation of DSAC has been carried out by using several methods in stage one, date seeds were washed by using distilled water to remove any impurities dried for 20 hours at 100 °C, and were crushed by using a pestle and mortar. Then, the impregnation process was carried out by using a ratio (3:1) in which 300 ml of 0.1 M KOH was used to soak 100 g of date seeds for 2 hours. Then were dried again before entering the furnace. The date seeds sample was dried in the furnace for 1 hour at 900 °C. Then, the date seed was treated by using 0.1 M HCl and distilled water to neutralize and remove excess chloride in the sample [5]. Lastly, date seeds were dried in the oven for 20 hours at 100 °C which produced date seed activated carbon (DSAC).

2.3 Characterization of DSAC

This study was preparing and producing date seeds activated carbon (DSAC) using chemical activation techniques. Produced DSAC has been characterized by using two instruments SEM and FTIR analysis. It is because want to analyze the surface morphology and functional group that is present in DSAC.

2.4 Adsorption of DSAC

The adsorption process was done by using 4 different dosages (10 g, 20 g, 30 g, and 40 g) of DSAC to treat 100 ml of 100 mg/L methylene blue. Then, 4 different treated samples were analyzed to determine their color, COD, and turbidity. These types of analyses are used to assess the DSAC's capability to treat methylene blue using the adsorption technique.

2.4.1 Color Removal Test

After the methylene blue solution has been treated with DSAC, the solution will be tested using DR 6000 spectrophotometer to determine the concentration of treated methylene blue.

$$CL\% = \frac{Cl_B - Cl_T}{Cl_B} \times 100\% \tag{1}$$

(1) will be used to compute the percentage of color removal (CL%) from the methylene blue sample where CI_B refers to blank (100 mg/L methylene blue) and Cl_T refers to the treated methylene blue.

Penerbit UTHM

2.4.2 Chemical Oxygen Demand (COD) Test

COD measurements were taken at 600 nm with a DR 6000 spectrophotometer. Batch tests (blank, 100 mg/L methylene blue, and varied solutions treated with DSAC) were performed to measure the COD. Experiments were conducted using an orbital shaker at 200 rpm for 180 minutes of contact time. Before effluent separation, each batch experiment's mixture was allowed to stand for 60 minutes without agitation. The COD concentrations in the filtrates were then determined [6].

2.4.3 Turbidity Test

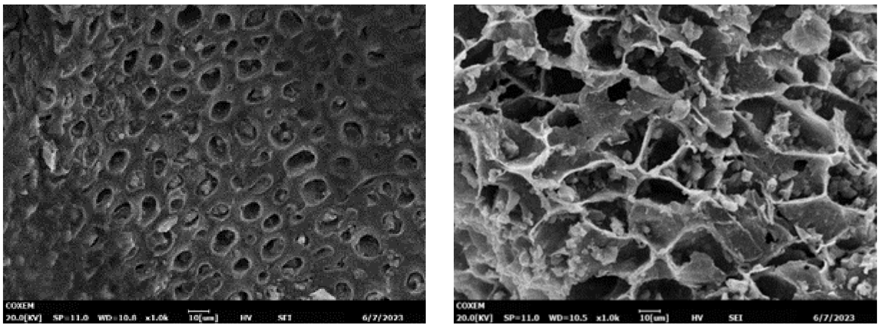
The turbidity of the methylene blue sample was measured using a DR 6000 spectrophotometer at 450 nm. In this investigation, a turbidity test has been utilized to determine the cloudiness (transparency) of the DSAC-treated solution. The NTU unit is used to represent the result of this turbidity meter.

3. Results and Discussion

3.1 Characterization of DSAC

3.1.1 SEM Analysis

SEM analysis has been used to determine the surface morphology of the activated carbon produced by date seed. Fig. 1 (a) shows the result of the surface shape of DSAC that has been captured by SEM with ×1000 magnification while Fig. 1 (b) with ×1500 magnifications. It shows the cavities hole that appear on the surface of DSAC. These pores of DSAC will be the main factor for the adsorption of methylene blue treatment. These pores were obtained after the impregnation method was used [7]. This indicates the pores on the activated carbon and increases the surface area when treated with chemicals (KOH and HCl). It can be concluded that this DSAC can be absorbent for the treatment of methylene blue. This is because the pores will attract to the particle of methylene blue solution and this process will "filtrate" the solution.



(a) **(b)**

Fig. 1 *SEM images of DSAC (a) ×1000 magnification; (b) ×1500 magnification*

3.1.2 FTIR Analysis

Fig. 2 shows the spectra from the analysis collected the functional groups for the DSAC samples using Fourier-Transform Infrared. The obtained FTIR spectrum revealed the peak at 3548 cm^{-1} related to the hydroxyl group's (O-H) stretching bands. The peak at 3250 and 2930cm^{-1} correspond to the presence of (-CH_3-) and (-CH_2-) group symmetric and asymmetric C-H stretching vibrations. CO_2 is observed based on the value 2568 cm^{-1}, thus is concluded that CO_2 is present in the air. Meanwhile 2162, 2024, and 1987 cm^{-1} value show C-H weak band aromatic from organic compounds. This shows that some organic compounds are still found in activated carbon. Other values like 3899, 3832, and 3705cm^{-1} and the value of 552cm^{-1} in the fingerprint region are not taken. Those values are noise caused by unwanted fluctuation in a signal. Fig. 2 is slightly error and different from other FTIR spectrum analyses because of the over-the-range value. However, it still can conclude the value by referring to the FTIR spectrum table and reference. Therefore, it shows that the functional groups observed in the DSAC during analysis were the most successful way to handle methylene blue.

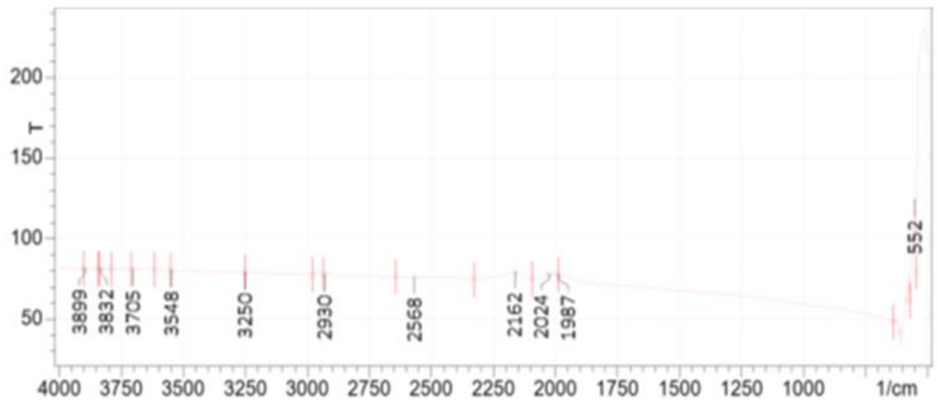


Fig. 2 *FTIR spectrum for DSAC*

3.2 Adsorption Efficiency

3.2.1 Color Removal Analysis

The efficacy of DSAC to remove color from the treated methylene blue solution was studied using various DSAC dosages. Fig. 3 depicts the produced graph of the CL% against DSAC dosage. When 10 g of DSAC is applied, it could decrease the color of methylene blue by 93.68%, whereas 20 g of DSAC reduces the color of methylene blue by 96.13%. When 40 g of DSAC was used to treat methylene blue, it reduced 96.61% of color. However, 30 g of DSAC, just reduces color for 88.56% of pure methylene blue. It supposedly removes color in between the range of 96.13% to 96.61%. The trendline demonstrates that a greater amount of DSAC may reduce the color of methylene blue.

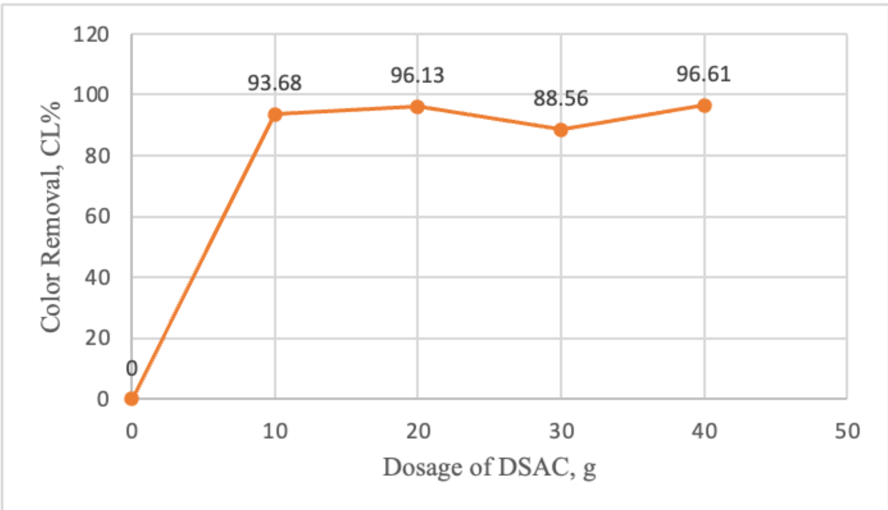


Fig. 3 *Relationship between the variable dosage of DSAC with percentage color removal*

3.2.2 COD Analysis

Table 1 shows the result of the COD parameter value for blank/untreated and treated methylene blue. 0 g dosage means the untreated methylene blue because it is not treated by using DSAC. Then, the value of COD for 10 g, 20 g, 30 g, and 40 g of DSAC were treated methylene blue.

As shown in Table 1, 10 g of DSAC is used to treat 100 ml of methylene blue, and the COD is increased from 109 mg/L to 364 mg/L. This also happens to 20 g, 30 g, and 40 g of DSAC used to treat methylene blue the COD values are 779 mg/L, 1109 mg/L, and 1416 mg/L. It shows when the amount of DSAC to treat methylene blue solution increases, the COD Percentage also increases. This shows that different DSAC dosages can increase methylene blue's COD value. Table 1 also shows percentage increases because DSAC did not remove the COD value below than untreated solution. As in theory, DSAC can reduce the COD value of methylene blue effectively but, in this study, it increases the value. It might be because of errors that occur during the preparation of DSAC. Thus, will increase the COD value of methylene blue after being treated.

Table 1 *COD value for 5 samples treated methylene blue*

Dosage of DSAC (g)	COD (mg/L)	COD Percentage Increase (%)
0	109	0.00
10	364	70.05
20	779	86.01
30	1109	90.17
40	1416	92.23

3.2.3 Turbidity Analysis

The turbidity value for treated methylene blue has been measured by using a DR 6000 Spectrophotometer. Table 2 shows data obtained between the dosage of DSAC and turbidity value.

Table 2 *Turbidity value for 5 samples treated methylene blue (0 g, 10 g, 20 g, 30 g and 40 g)*

Dosage of DSAC (g)	Turbidity value, NTU
0	1.55
10	2.32
20	3.05
30	10.16
40	3.90

According to Table 2, the DSAC dosage can increase the turbidity value after the adsorption process has been done. 10 g of DSAC can increase the turbidity from untreated methylene blue which from 1.55 NTU to 2.32 NTU. This trendline also occurs in other dosages of DSAC which 20 g increased the turbidity to 3.05 NTU while 40 g increased it to 3.90 NTU. However, there is an error occurs when 30 g of DSAC used to treat 100 ml of methylene blue. It increases the turbidity value to 10.16 NTU. These results show the increased turbidity after being treated by DSAC is due to an excess of DSAC that is used to treat methylene blue. The excess of DSAC in treated methylene blue can cause the interference of light when it passes through the treated methylene blue. This phenomenon will affect the turbidity value. To overcome this issue, it must undergo a centrifuge process to remove all the excess after the treated solution has been filtered.

4. Conclusion

In this study, the adsorption of methylene blue using date seed-activated carbon has shown a positive result in terms of removing color. The objective of this study is to determine which DSAC can reduce the color of methylene blue, unfortunately, not for COD and turbidity removal parameters. This shows that DSAC can be used as an effective absorbent since when increasing the amount of DSAC used, it can reduce color more effectively. However, DSAC can harm COD and turbidity value which increases both parameters. This is because the effect on DSAC is contaminated by organic and inorganic material before the adsorption process occurs. When preparing activated carbon, it must treat the sample carefully to make sure that it removes all the excess chloride that came from KOH and distillate the sample using distilled water. For turbidity and COD analysis, using massive amount of DSAC like its state in the method process can harm the reading. The consequence that will be are the reading is higher than it should be. The chosen dosage was based on the scope of the past project. The recommendation for improvement is to use an appropriate dosage for the next research.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Muhammad Fauzan Syafi Mohamed Foudi, Mohd Nur Aiman Syazani Huzairi, Raudah Mohd Adnan, Dilaeleyana Abu Bakar Sidik;* ***data collection:*** *Muhammad Fauzan Syafi Mohamed Foudi, Mohd Nur Aiman Syazani Huzairi, Raudah Mohd Adnan, Dilaeleyana Abu Bakar Sidik;* ***analysis and interpretation of results:*** *Muhammad Fauzan Syafi Mohamed Foudi, Mohd Nur Aiman Syazani Huzairi, Raudah Mohd Adnan, Dilaeleyana Abu Bakar Sidik;* ***draft manuscript preparation:*** *Muhammad Fauzan Syafi Mohamed Foudi, Mohd Nur Aiman Syazani Huzairi, Raudah Mohd Adnan, Dilaeleyana Abu Bakar Sidik. All authors reviewed the results and approved the final version of the manuscript.*

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