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PRODUCTION OF ACTIVATED CARBON OF PALMYRA PALM FRUIT SHELL (*Borassus flabellifer* Linn.) FROM KISAR ISLAND, SOUTHWEST MALUKU REGENCY FOR ADSORPTION OF METHYL RED DYE

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ABSTRACT

Palmyra palm is a species of palmae that grows a lot on Kisar Island where all parts of the plant can be utilized. However, the palmyra palm fruit shell has not been used and that is only thrown away. In this study, palmyra palm fruit shell were made as activated carbon and were characterized using XRD to determine the crystallinity properties of the palmyra palm fruit shell activated carbon and adsorption test was carried out to determine the capacity and efficiency of activated carbon adsorption to methyl red dye. The production of palmyra palm fruit shell activated carbon is carried out through the carbonization stage at 300°C for 1 hour, and followed by chemical activation using H_3PO_4 10% solution for 24 hours. The results of the analysis using XRD showed that the activated carbon of the palmyra palm fruit shell formed peaks with the highest intensity at diffraction angles of 2θ = 26.5241° and 27.6274° with d-spacing values (Å) = 3.36059 and 3.22884, which are characteristic peaks of carbon and are amorphous. The adsorption of methyl red 20 ppm use batch method, with various contact times of 5, 10, 15, 20, 25, and 30 minutes. The results of showed that on 15 minutes was the optimal time, with 0.25 gram mass of the adsorbent and the adsorption capacity and adsorption efficiency was 0.87 mg/g and 44.3%, respectively.

Keywords: *Activated Carbon, Palmyra Palm Shell, Methyl Red, Adsorption*

INTRODUCTION

The development of the textile industry in Indonesia is currently experiencing very rapid production growth from year to year. This development provides many benefits for human life, besides that it also has a negative impact on the environment (Lestari, 2020). The textile industry produces dye waste which will be discharged into water bodies. Disposal of textile waste into water bodies has caused a lot of concern because of the potential health hazards associated with the entry of toxic components into the human and animal food chains (Dim, 2013), besides that it can also cause an increase in BOD and also cause water to have a high level of color (Rachmawati et al, 2022).

In the textile industry, the use of dyes is very large (Akinola & Umar, 2015). Dyes are a non-biodegradable pollutant, generally made from azo compounds and their derivatives which are benzene groups. Around 60-70% of azo compounds are used in the textile industry because they are cheap, easy to synthesize and the stability and availability of various colors compared to natural dyes (Permatasari et al, 2018). Textile dye waste must be treated before being discharged into water bodies, because around 10% to 15% of dyes that have been used cannot be reused and must be disposed of. If azo compounds are present in the environment for too long, they will become a source of disease because they are carcinogenic and mutagenic. According to the Regulation of the Minister of Environment of the Republic of Indonesia Number 5 of 2014 concerning Liquid Waste Quality Standards for Industrial Activities, azo group compounds have a threshold of 5 mg/L. One of the azo dyes that is widely used in the dyeing process is methyl red (Lestari, 2020).

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Methyl red has an azo group chromophore system (-N=N-) which binds to an aromatic group, is a synthetic dye and is the most reactive in the dyeing process of textile materials (Laksono, 2009). These dyes are used in dyeing fabrics, laboratory testing, textiles, and other commercial products, but may cause eye and skin sensitization and irritation of the pharynx or digestive tract if inhaled or ingested. Furthermore, it can be mutagenic under aerobic conditions (Ahmad et al, 2015).

Most of the conventional wastewater treatment uses biological processes, while textile dyes have aromatic structures, especially reactive dyes which are difficult to decompose by biological wastewater treatment systems (Purnama & Kurnianto, 2016). In addition, biological processing methods require quite expensive costs, especially in operation. Wastewater treatment by adsorption process is an easy and inexpensive way because of the low initial cost, flexibility, simplicity of design, and ease of operation to treat textile industry wastewater (Isiuku & Onyema, 2017).

In the adsorption process required an appropriate adsorbent. The use of cheap and environmentally friendly adsorbents is necessary to reduce the cost of the adsorption process. One of the potential adsorbents to be developed is activated carbon (Kurniawan et al, 2012). Activated carbon is one of the materials that can be used to treat environmental pollutant waste and is the best adsorbent in adsorption systems, because it has a large surface area and high adsorption power so that its utilization can be optimal (Nitsae et al, 2020). The presence of functional groups found in activated carbon adsorbents such as –OH (Hydroxyl), C=O (Carbonyl), -COOH (Carboxyl), and aromatic C-H can be used as dye absorbents (Ihsan, 2022). In general, activated carbon can be made using coal and materials containing ligno cellulose as raw materials. Sources of cellulose that can be used include remnants of agricultural products and forest products, used paper, and industrial waste (Agusdin & Setiorini, 2020).

Palmyra palm (*Borassus flabelifer* L.) is a type of palm or Arecaceae which grows mainly in dry areas. This plant can grow well in rocky and barren areas such as on Kisar Island, Southwest Maluku (Sahusilawane et al, 2018). Astronomically, Kisar Island is located at 8.06° South Latitude and 127.18° East Longitude with an area of 177.59 km2. All parts of the palmyra palm plant are used, namely leaves, stems, fruit and flowers for the needs of the local community. While the palmyra palm fruit shell is wasted as waste that is rarely used and only thrown away. Palmyra palm fruit shell contains 11.90% cellulose, 13.80% water, 44.58% carbon, 4.46% ash, and 23.85% volatile matter which has the potential to be the main ingredient for making activated carbon (Anggraeni & Yuliana, 2015).

Based on the explanation above, the researcher is interested in conducting research with the title: "Production of activated carbon of palmyra palm fruit shell (*Borassus flabelifer* Linn.) from Kisar Island, Southwest Maluku Regency for the adsorption of methyl red dye".

RESEARCH METHODS

The materials used were Palmyra palm fruit shell taken from Kisar Island MBD, Methyl red, Aquades, Whatman No. filter paper. 42, 10% H_3PO_4 Solution.

1. Production of Activated Carbon from Palmyra Palm Shell
   a. Dehydration
   Palmyra palm shells are taken from fruits that are old and hard, washed and cleaned of the husks and adhering fruit, then dried in the sun to remove the water still contained in them. The shells were weighed until a constant weight was obtained.
   b. Carbonization
   The carbonization stage follows the procedure carried out by Anggraeni & Yuliana (2015) with modifications according to research needs, namely 460.32 g of palmyra palm fruit shells are put into the furnace at 300ºC for 1 hour, then the charcoal is cooled, ground and filtered through a 100 mesh sieve to get the same particle size.
   c. Activation
   The activation stage follows the procedure carried out by Khuluk & Rahmat (2019) with a modification, namely the activation is carried out using a 10% H_3PO_4 acid activator. The ratio of the volume of activator to the mass of carbon powder is 4:1, namely 200 mL of H_3PO_4 and 50 g of carbon. The mixture was stirred for 24 hours at 300 rpm. Then the mixture was filtered using Whatmann No. filter paper. 42

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and washed with distilled water until the pH is neutral. Next, it is dried in an oven at 110ºC for 5 hours. After that it was cooled in a desiccator.

2. Characterization of Palmyra Palm Fruit Shell Activated Carbon
Palmyra palm fruit shell activated carbon obtained was then characterized using X-Ray Diffraction to identify the crystalline phase in the material.
3. The Study of Methyl Red Adsorption with Variation of Contact Time
A total of 0.25 g of activated carbon was put into the Erlenmeyer. Then 25 mL of 20 ppm methyl red solution was added. Adsorption is carried out with a Batch system and the mixture is stirred. The contact time was varied for 5, 10, 15, 20, 25 and 30 minutes. Then filtered using filter paper. The absorbance of the filtrate was measured using a UV-Vis spectrophotometer at a wavelength of 509.8 nm and determined using a standard curve (Nurkholifa, 2019).

Data analysis

The data that has been obtained is the absorbance of the methyl red solution after being adsorbed at various contact times. Then enter into the regression equation, namely y = ax + b, where a is the slope, b is the intercept, x is the concentration and y is the absorbance. So that it can be determined the concentration of the solution after being adsorbed. Contact time variation data is then used to determine adsorption capacity and adsorption efficiency. Adsorption capacity and efficiency can be calculated using equations 1 and 2.

$$q_t = \frac{C_0 - C_e}{m} \times V \quad \text{...(1)}$$

Where q_t = Adsorption capacity per molecular weight (mg/g), C_0 = Initial concentration of solution (mg/L), C_e = Concentration of solution after adsorbed (mg/L), V = Dye solution volume (L), and m = Adsorbent mass (g).

$$\text{Adsorption efficiency (\%)} = \frac{C_0 - C_e}{C_0} \times 100\% \quad \text{...(2)}$$

Where C_0 = Initial concentration of solution (mg/L), and C_e = Concentration of solution after adsorbed (mg/L).

RESULTS AND DISCUSSION

A. Palmyra Palm Fruit Shell Activated Carbon

The sample used in this study was palmyra palm fruit shells (*Borassus flabellifer* L.) obtained from Kisar Island, Southwest Maluku Regency. The production of activated carbon is carried out in three stages, namely dehydration, carbonization and activation. In the initial treatment, the palmyra palm fruit shells were washed and cleaned of coir and attached fruit. The shells that have been cleaned are then subjected to a dehydration process, namely removing the water content contained in the palmyra palm fruit shells by drying the shells in the sun until a constant weight is obtained.

The dried palmyra palm fruit shells were then cut into small pieces with the aim of reducing the size so that the carbonization process was more even. The smaller the size of the shell, the greater the surface area of the contact area of the shell which is in contact with heat during the carbonization process (Yuliusman in Nurkholifa, 2019). Carbonization was carried out at 300°C for 1 hour using a furnace. At this temperature, hemicellulose, cellulose and lignin decompose in gaseous forms such as H_2, CH_4, tar, methanol and hydrocarbons, so that the palmyra palm fruit shell slowly turns into charcoal (Anggraeni & Yuliana, 2015). The carbonization process emits a lot of smoke which indicates that the volatile compounds contained in the palmyra palm fruit shell have evaporated (Nurkholifa, 2019). Carbonization results obtained 132.18 g of carbon (28.71%). The carbon that has been produced from the carbonization process is chemically activated using 10% H_3PO_4. Chemical activation is used because it has several advantages compared to physical activation such as the activation temperature used is relatively low and the pores are formed more so that the surface area is large (Asrijal et al, 2014). H_3PO_4 is used as an activator because it is a very strong dehydrating agent which has $3H^+$ which is able to push non-volatile substances that are still left behind so that the pores in activated carbon get bigger and the wall structure is stronger. In the activation process H_3PO_4 will be adsorbed by carbon which will dissolve tar compounds and impurities. Tar is a polluting hydrocarbon compound left over from combustion in the carbonization process which

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will cover the pores of activated carbon. The loss of these substances from the surface of activated carbon will cause the pores to open more. The wide pores of activated carbon can increase the surface area of activated carbon so that it will increase the adsorption ability of the activated carbon (Jacob, 2021). H_3PO_4 also does not pollute the environment and the process of neutralizing active carbon products is easy, that is, only by washing with water. Palmyra palm fruit shell activated carbon is shown in **Figure 1.**

Figure 1. Palmyra Palm Fruit Shell

B. Characterization of Palmyra Palm Fruit Shell Activated Carbon Using X-Ray Diffraction (XRD)

The activated carbon that has been produced is characterized using X-Ray Diffraction to identify the crystalline phase and crystalline structure in the material. Determination of the crystalline structure of activated carbon aims to determine the presence of highly ordered (crystalline) and irregular (amorphous) crystal structures. The results of the XRD characterization are in the form of a diffraction pattern with a relationship of 2θ to the scattering intensity with measurements at an angle range of 5°- 60°, as shown in **Figure 2.**

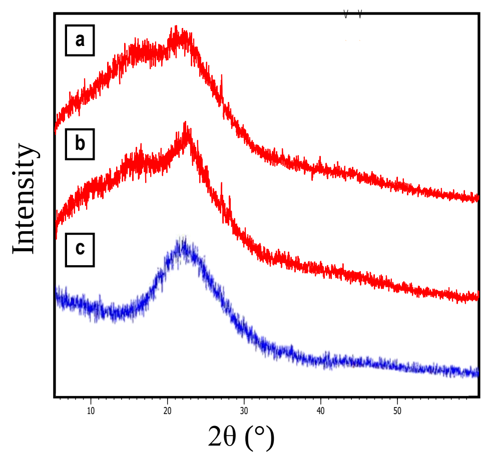

Figure 2. XRD diffractogram (a) Study Sample of Palmyra palm fruit shell carbon (b) Study sample of Palmyra palm fruit shell activated carbon(c) Rice husk carbon (Solihudin et al, 2020)

Figures 2a and 2b show a widened diffractogram peak. The wide peak at 20°-30° is a characteristic peak for carbon with a low degree of crystallization, indicating that the sample has an amorphous surface structure with a heterogeneous surface (Mulyati, 2017). From figure 2(a), it can be seen that the peak is at a diffraction angle of 2θ = 26.6001° with a d-spacing value (Å) = 3.34839. From figure 2(b) forms peaks at diffraction angles 2θ = 26.5241° and 27.6274° with d-spacing values (Å) = 3.36059 and 3.22884. The resulting activated carbon

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diffraction pattern is compared to rice husk carbon in the study of Solihudin, et al (2020) in Figure 2c. From the carbon diffractogram, it shows that the peak shape is not sharp and the wide angle range indicates that the carbon produced is amorphous. Islam, et al (2016) also reported the same diffractogram pattern using coconut shell activated carbon showing peaks with high intensity at diffraction angles of 2θ = 26° and 42° having an amorphous diffraction pattern.

Based on the results of the analysis with XRD, it can be concluded that the activated carbon of palmyra palm fruit shell is amorphous. The amorphous state is a state or phase in which the arrangement of the atoms formed changes in the regularity of the constituent particles. Amorphous is formed due to the cooling process that is too fast so that the atoms cannot properly occupy the lattice location.

C. Methyl Red Adsorption Using Palmyra Palm Fruit Shell Activated Carbon

In this research, the contact time variation was carried out between the adsorbent and the adsorbate to determine the optimum time in the adsorption process. Contact time is one of the factors that affect the amount of adsorbate to be adsorbed. The color that decreases with increasing contact time indicates that the adsorbent has successfully absorbed the adsorbate. After varying the time the adsorption mixture was filtered using filter paper, the filtrate was collected and the absorbance was measured using a UV-Vis spectrophotometer. Data on the results of measuring the absorbance of the methyl red solution after being adsorbed at various contact times are shown in **Table 1**.

Table 1. Concentration and Absorbance of Methyl Red Solution After Adsorption Process Using Activated Carbon with Variation of Contact Time

Contact Time (Minute)	After Adsorption		Adsorption Capacity (mg/g)	Adsorption Efficiency (%)
	Absorbance	Concentration (ppm)		
5	0,094	11,84	0,78	39,61
10	0,089	11,18	0,84	42,97
15	**0,087**	**10,92**	**0,87**	**44,3**
20	0,092	11,58	0,802	40,93
25	0,104	13,16	0,64	32,87
30	0,106	13,42	0,62	31,55

The initial concentration of methyl red is 19.605 $mg.L^{-1}$. The concentration of methyl red after the adsorption process can be determined by entering the absorbance data into the regression equation obtained from the standard curve of methyl red solution, namely $y = 0.0076x + 0.004$.

The results of the analysis in Table 1 show that the optimum adsorption of activated carbon occurs in 15 minutes with an adsorption capacity of 0.87 mg/g and an adsorption efficiency of 44.3%. The increase in adsorption occurred from 5 minutes to 15 minutes. This is due to the more interactions and collisions that occur between the activated carbon of the palmyra palm fruit shell and methyl red as the interaction takes longer. In the 5 minutes the adsorption of methyl red occurs quickly because the pores of the activated carbon have not interacted much with the adsorbate, making it easier for the adsorbate to fill the pores (Kustomo & Santosa, 2019).

The optimum adsorption efficiency of palmyra palm fruit shell activated carbon for methyl red was lower than the activated carbon of Annona squmosa seeds as reported by Shanti, et al (2010) with a contact time of 100 minutes of 82.81%. This is due to differences in the initial concentration of methyl red, the characteristics of the activated carbon produced, as well as differences in other variables that affect the adsorption process. Goa, et al (2014) reported the use of palmyra palm fruit shell activated carbon to adsorb Mn and Fe metals in manganese washing waste with adsorption efficiencies of 66.46% and 77.51% respectively. The low adsorption efficiency obtained is associated with many of the carbonized residues that are non-porous (Kayadoe et al, 2020).

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At a contact time of 20 minutes the adsorption capacity and efficiency decreased to 0.802 mg/g and 40.93%. The decrease in adsorption capacity continued at 25 and 30 minutes. This decrease is due to desorption or release of the adsorbate again during stirring (Nurkholifa, 2019). Desorption occurs because the surface of the adsorbent is saturated or is no longer able to absorb the adsorbate and an equilibrium occurs so that the methyl red dye which was initially adsorbed by the adsorbent will be released again (Fuadah & Rahmayanti, 2019).

D. Adsorption mechanism

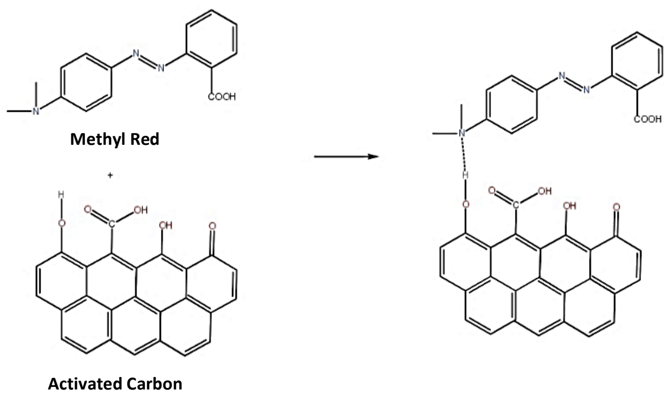

Figure 2. Mechanism of Adsorption of Activated Carbon with Methyl Red (Yuan et al, 2019)

Several possible adsorption mechanisms including electrostatic interactions, hydrogen bonds, have been proposed to understand the interactions between solid adsorbents and dyes in aqueous solutions. In this study, methyl red was adsorbed onto the surface of activated carbon through electrostatic interactions. The O-H group on the surface of activated carbon can also interact with the amine group of the red methyl molecule through hydrogen bonds (Yuan et al, 2019).

CONCLUSION

1. The results of the characterization of palmyra palm fruit shell activated carbon using XRD show characteristic peaks of carbon with a low degree of crystallization, sharp peak shapes and a wide angle range indicating that the resulting palmyra palm fruit shell activated carbon is amorphous.
2. The optimum adsorption capacity and adsorption efficiency of activated carbon of palmyra palm fruit shell on methyl red dye at a concentration of 20 ppm was at a contact time of 15 minutes, with an adsorbent mass of 0.25 g, respectively 0.87 mg/g and 44, 3 %.

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