

Utilization of Coffee Grounds as an Adsorbent in Reducing the Levels of Naphthol Dyes of Weaving Industry Wastewater

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ABSTRACT

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Naphthol dye is one of the synthetic dyes contained in weaving industry waste. One method that can be used to reduce the disadvantageous effects of these substances is by adsorption using activated charcoal adsorbent from coffee grounds. This research aims to determine the character of activated charcoal from coffee grounds and the effect of adsorbent dosage and stirring time on the adsorption of naphthol dye levels. Making activated charcoal from coffee grounds goes through 3 stages, namely the dehydration stage, carbonization stage at 350 °C for 3.5 hours, and activation stage using a 0.1 M HCl activator for 48 hours. The characterization of activated charcoal obtained complies with SNI 06-3730-1995 with results of water content of 3.74%, ash content of 2.12%, and iodine adsorption capacity of 758.29 mg/g, and functional group testing was carried out by varying the adsorbent dosage at 0.125; 0.250; 0.500; 0.750; 1 g (per 100 mL sample) and stirring time for 10; 20; 30; 40; 50 minutes. From the research results, it was obtained the best adsorbent dose and mixing time were 0.5 g and 40 minutes with adsorption efficiency of 98.68% and 99.63%.

Keywords: coffee grounds; active charcoal; weaving waste; adsorption; naphthol

1. INTRODUCTION

The weaving industry is a textile industry that produces traditionally woven cloth crafts using non-machines. The process of making woven fabric produces liquid waste reaching \pm 1000 liters/day, many of which contain synthetic chemical dyes such as naphthol and indanthrene dyes. Naphthol dye is a type of artificial dye that can be used to dye materials quickly and produce intense colors. The naphthol dye consists of two basic components that are not soluble in water, namely the naphthol group AS (anilid acid) and the color-generating component which is also known as the diazonium or salt group [1]. The liquid waste produced from the remainder of the woven fabric production process is immediately thrown into the gutter without being treated first, which causes water pollution around the weaving industry.

The adsorption method is one method for removing pollutants from wastewater. It is called adsorption because it can absorb ions or molecular substances on the surface of the adsorbent [2] (Syauqiah et al., 2011). Activated charcoal is a type

of adsorbent that is quite effective in absorbing dyes [1]. Activated charcoal can be produced from natural materials such as coffee grounds, which can be made into activated charcoal because it has a total carbon content of 47.8-58.9%, total nitrogen of 1.9-2.3%, protein of 6.7- 13.6%, ash 0.43-1.6% and cellulose 8.6% [3]. The relatively high carbon content in coffee grounds means that coffee grounds waste can be used to make activated charcoal.

2. MATERIALS AND METHODS

2.1 Materials

The tools used are analytical balance, sieve shaker, furnace, oven, glass beaker, crushible, desiccator, pH meter, UV-Vis spectrophotometry, erlenmeyer, stirring rod, Whatman No. 42 filter paper, dropper pipette, measuring flask, and volumetric flask. The materials used are coffee grounds, HCl, distilled water, weaving industry liquid waste, naphthol dyes, NaOH, and H₂SO₄.

2.2 Method of activated charcoal

Making activated charcoal from coffee grounds goes through 3 stages, namely (a) the coffee grounds dehydration process using an oven at 105°C for 2 hours, (b) the coffee grounds carbonization process using a furnace at 350°C for 3.5 hours, then the resulting charcoal is screened using a 100 mesh screen, and (c) the coffee grounds charcoal activation process uses a hydrochloric acid (HCl) activator for 48 hours. Next, the activated charcoal is washed using distilled water until the pH is neutral and dried using an oven at 105°C until the weight is constant. The resulting activated charcoal was characterized and analyzed. The reduction in color content was carried out using 100 ml of wastewater with the pH adjusted to 4. Then the adsorption process was carried out using varying adsorbent doses of 0.125; 0.250; 0.500; 0.750; and 1 gram, with a stirring time of 30 minutes. The best results from varying adsorbent doses were used for the adsorption process using varying stirring times of 10; 20; 30; 40; and 50 minutes. Analysis of the results of the adsorption process was carried out to determine the effect of adsorbent dosage and stirring time [4] [5].

2.3 Analysis of activated charcoal

2.3.1 Water content

Water content analysis was carried out by weighing 1 gram of activated charcoal and heating it in an oven at 115°C for 3 hours. Next, the activated charcoal is put into a desiccator and then weighed until the weight is constant.

2.3.2 Ash content

Ash content analysis was carried out by weighing 1 gram of activated charcoal and burning it in a furnace at a temperature of 650°C for 2 hours. Next, the activated charcoal ash is put into a desiccator and weighed until the weight is constant.

2.3.3 Iodine Absorption Capacity

Analysis of iodine absorption capacity was carried out by weighing 1 gram of activated charcoal and mixing it with 25 ml of 0.1 N iodine solution. Then stirring for 15 minutes, 10 ml of the filtrate was taken and titrated using 0.1 N sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) solution until it turned faint yellow, then titrated again by adding 1% starch solution as an indicator, and titrated until the blue color disappears or becomes clear.

2.4. FTIR test

The FTIR test was carried out to determine the structure of charcoal, activated charcoal, and activated charcoal after the adsorption process. The FTIR test was carried out using a Shimadzu IR Tracer-100 type FTIR tool.

2.5 Data Analysis

In this research, the statistical data analysis used was a One-Way ANOVA analysis. Through this analysis, a P-value will be obtained with a confidence level of 95% or $\alpha = 0.05$. Then, followed by Duncan's post-hoc analysis to find out where the differences in results come from. Post-hoc tests are used after finding statistically significant results. Statistical analysis was carried out using the SPSS version 27 program for MacOS.

3. RESULTS AND DISCUSSION

This research was carried out to determine the characteristics of activated charcoal from coffee grounds, determining the effect of adsorbent dosage and stirring time on reducing dye levels in liquid waste from the weaving industry. Making activated charcoal from coffee grounds in this research goes through 3 stages, namely the first dehydration stage aims to remove the water content in the coffee grounds, the second carbonization stage is the process of burning coffee grounds which aims to produce charcoal, the third stage activates the charcoal using the chemical activation method. The activation stage aims to expand the pores in the charcoal using the help of an activator in the form of an acid or base solution, whereas in this study the activation stage uses an activator from a 0.1 M hydrochloric acid (HCl) solution. The reaction mechanism for charcoal with the hydrochloric acid (HCl) activator can be seen in **Figure 1**. The cellulose content in coffee grounds charcoal which contains quite a lot of oxygen makes hydrochloric acid (HCl) the right choice to be used as an activating agent. This is because when HCl is used as an activator, the HCl will react with the charcoal by binding to oxygen levels and then damaging the inside of the charcoal until larger pores are formed.

3.1 Characterization of Activated Charcoal from Coffee Grounds

Activated charcoal from coffee grounds that have gone through the chemical activation stage requires characterization of the activated charcoal content obtained. Activated charcoal characterization aims to determine whether the activated charcoal from coffee grounds is following SNI 06-3730-1995 or not. The results of the characterization of charcoal and activated charcoal from coffee grounds based on the parameters of water content, ash content, and iodine absorption capacity are listed in **Table 1**.

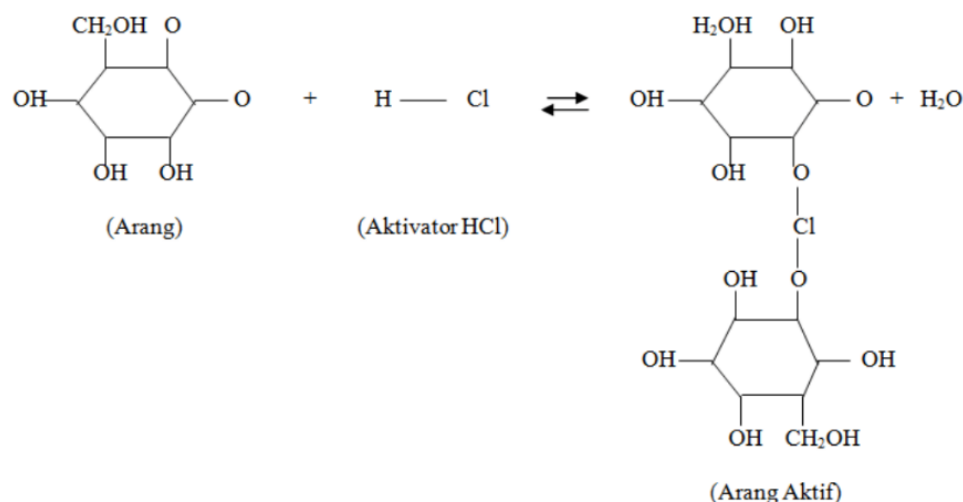


Figure 1 Reaction mechanism of activated charcoal activated with HCl (Safitri, 2016)

Table 1 Characterization of charcoal and activated charcoal from coffee grounds

Parameter	Test Result		SNI 06-3730-1995
	Charcoal	Activated Charcoal	
Water content	4.62%	3.74%	Maximum 15%
Ash content	6.67%	2.12%	Maximum 10%
Iodine adsorption capacity	342.33 mg/g	758.29 mg/g	Minimum 750 mg/g

3.2 Moisture Content Characterization of Activated Charcoal from Coffee Grounds

Water content testing aims to obtain hygroscopic properties of activated charcoal from coffee grounds. The water content value can indicate high or low water lining the pores of the activated charcoal [1]. Based on the test results, the water content in charcoal from coffee grounds was 4.62% and the water content in activated charcoal from coffee grounds was 3.74%. This shows that the water content obtained has met the criteria of SNI 06-3730-1995, namely the water content of activated charcoal from coffee grounds is still below the maximum limit of 15%. This happens because the dehydration and carbonization process has exceeded the temperature of 100°C, so chemically the water molecules that are heated above their boiling point change phase from liquid to gas [6].

The use of HCl as an activator helps increase efficiency in reducing the water content of the activated charcoal produced. The water content obtained by activated charcoal from coffee grounds is lower than charcoal that has not been activated,

this shows that the water content contained in activated charcoal from coffee grounds after being activated by HCl has decreased. The fewer water molecules there are in activated charcoal, the fewer obstacles there are for other molecules to enter it [7].

3.3. Ash Content Characterization of Activated Charcoal from Coffee Grounds

Ash content testing is aimed at obtaining information regarding the metal oxide content or inorganic content contained in activated charcoal. Ash content is defined as the remaining minerals that are unable to evaporate during the carbonization process [8]. In this study, the results obtained were that the ash content in charcoal from coffee grounds was 6.67%, while the ash content in activated charcoal from coffee grounds was 2.12%. These results show that activated charcoal activated by hydrochloric acid (HCl) has a lower ash content compared to charcoal that is not activated. The activated charcoal obtained meets the criteria of SNI 06-3730-1995, namely the ash content in activated charcoal is still below the maximum limit of 10%. The

ash content value obtained shows that the metal oxide or inorganic content in the activated charcoal is relatively low. Ash content has a significant impact on the quality of activated charcoal. The higher ash content can cause blockage of the charcoal pores which results in a decrease in the surface area of the activated charcoal [8].

3.4 Characterization of Activated Charcoal Iodine Adsorption from Coffee Grounds

Iodine adsorption testing is aimed at obtaining information regarding the ability of activated charcoal to adsorb small-weight molecules in the liquid phase [3]. The characteristics of activated charcoal such as bound carbon content, ash content, water content, surface area and yield greatly influence the adsorption ability. However, the factor that most influences adsorption capacity is the surface area of activated charcoal, because the adsorption mechanism is closely related to the number of pores in the activated charcoal [9]. The results of the iodine adsorption test carried out on charcoal from coffee grounds were 342.33 mg/g, while on activated charcoal from coffee grounds, the result was 758.29 mg/g. This shows that coffee grounds activated charcoal activated by hydrochloric acid (HCl) produces a higher iodine adsorption capacity compared to the iodine adsorption capacity of unactivated charcoal. The activated charcoal obtained has met the criteria of SNI 06-3730-1995, namely the iodine adsorption capacity of activated charcoal has exceeded the minimum limit of 750 mg/g.

Based on research conducted by Alfiany et al. [10], activated charcoal that has been activated using hydrochloric acid (HCl) has a higher iodine adsorption capacity. This is caused by the activator effect of hydrochloric acid which reacts with the minerals in activated charcoal, forming salt compounds. This salt acts as a dehydrating agent and makes it easier to remove hydrocarbon deposits that have formed in the carbonization process. This makes the charcoal surface active and able to adsorb iodine effectively. The results of testing for water content, ash content, and iodine adsorption indicate that the activated charcoal produced in this research has characteristics that comply with SNI 06-3730-1995 and is capable of being used as an adsorbent in reducing the levels of naphthol dyes in liquid waste from the weaving industry.

3.5 Characterization of Fourier Transform Infra-Red (FTIR)

Fourier Transform Infra-Red (FTIR) characterization is an infrared spectroscopy instrument that aims to identify the presence of molecular functional groups contained in a sample. FTIR wave measurements are carried out using the infrared spectrum in the wavelength range 400-4000 cm^{-1} [11]. FTIR testing was carried out on three samples including coffee grounds charcoal, coffee grounds activated charcoal, and coffee grounds activated charcoal after the adsorption process. The FTIR test results for the three samples are listed in **Figure 2**.

The results of the FTIR characterization test on charcoal from coffee grounds, activated charcoal from coffee grounds, and activated charcoal from coffee grounds after going through an adsorption process on weaving industry wastewater. In the functional groups between coffee grounds charcoal and coffee grounds activated charcoal, there is no visible difference in functional groups. Based on research by Andamari [11], charcoal from coffee grounds activated using the HCl activator does not provide any differences in functional group bonds, but this activator can remove impurities contained in charcoal from coffee grounds and increase the surface area of the activated charcoal. At the wave peak of 1573.91 cm^{-1} of activated charcoal before the adsorption process and 1519.91 cm^{-1} of activated charcoal after the adsorption process, the C=O bond shows a decrease in wave peak absorption. This shows that the C=O bond is involved in the adsorption process of naphthol dye.

The wave peak of activated charcoal before the adsorption process has a peak wave number of 1219.01 cm^{-1} and activated charcoal after the adsorption process has a peak wave number of 1195.87 cm^{-1} . This shows that there is a carboxyl functional group (-COOH) which is at a wave range of 1100-1500 cm^{-1} [11]. In the vibration of the carboxyl functional group (-COOH), it was seen that there was a reduction in the carboxyl bond (-COOH) in activated charcoal after the adsorption process, which indicates that the carboxyl bond (-COOH) was involved in the absorption process of naphthol dye [12].

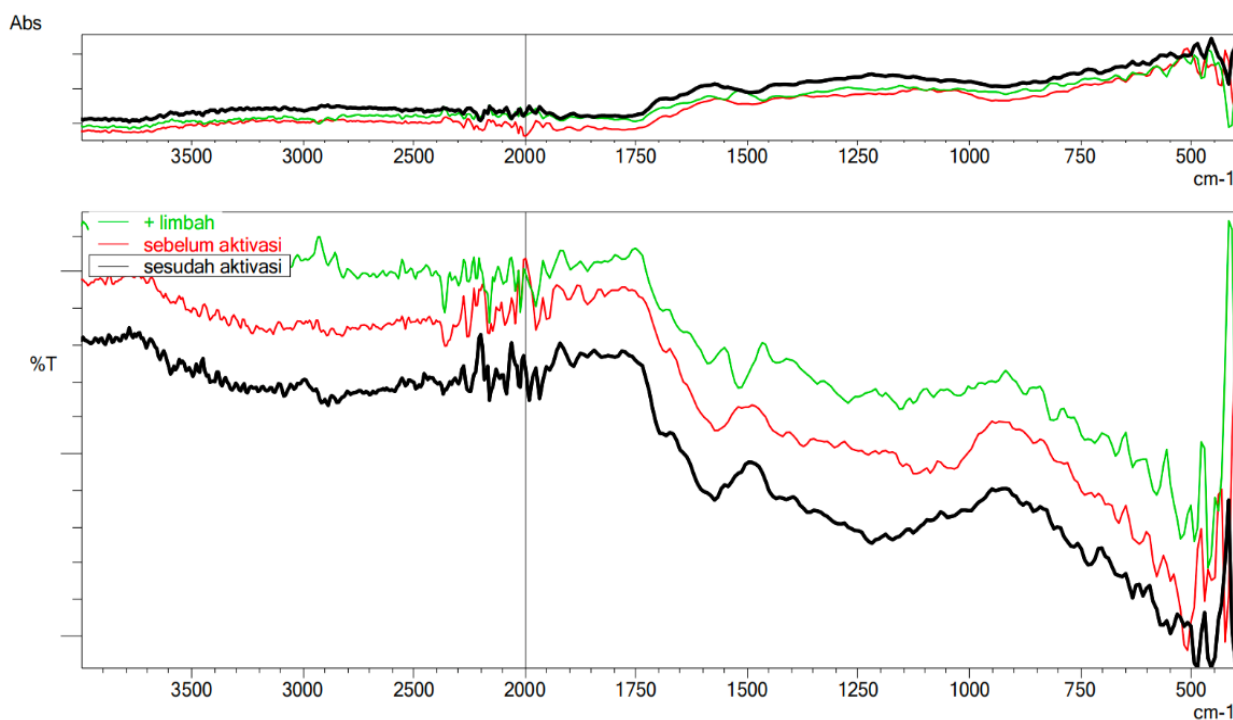


Figure 2 FTIR characterization test result

The results of FTIR characterization carried out on coffee grounds charcoal, coffee grounds activated charcoal, and coffee grounds activated charcoal after the adsorption process showed that the activated charcoal before the adsorption process and the activated charcoal after the adsorption process showed changes in the wave peaks. This indicates that contact has occurred between the adsorbed substance molecules and the functional groups contained in activated charcoal [12]

3.6 Effect of Adsorbent Dosage on Adsorption of Naphthol Dye Levels

Adsorbent dosage is a factor that can influence the adsorption process. The effectiveness of the adsorption process occurs when more doses of adsorbent are used. The effect of adsorbent dose on the absorption of naphthol dye in the weaving industry liquid waste can be determined by varying the adsorbent dose of activated charcoal from coffee grounds. This was done to determine the best adsorbent dose in the naphthol dye adsorption process by using varying adsorbent doses of 0.125; 0.250; 0.500; 0.750; and 1 gram and stirring time for 30 minutes. After the treatment of wastewater, the results obtained are listed in Table 2.

Table 1 Adsorption results of naphthol dye levels with varying adsorbent doses

Doses (grams)	Remaining dye levels (Ce)	Decrease in dye levels (%)
0.125	12.9782±0.09228 ^a	93.3578±0.04723 ^d
0.250	9.4348±1.29125 ^b	95.1713±0.66086 ^c
0.500	2.5870±0.33821 ^d	98.6760±0.17310 ^a
0.750	5.0652±0.46118 ^c	97.4076±0.23596 ^b
1.000	6.2565±0.18441 ^c	96.8180±0.09440 ^b

Note: Homogeneity data is marked with a superscript letter. The letters 'a' to 'd' indicate the highest value to the lowest

Based on the data in Table 2, increasing the adsorbent dosage from 0.125 to 0.500 significantly (P -value < 0.05) affects the remaining dye content and the percentage of color removal in weaving industry wastewater. The best dose occurred at 0.500 grams with a remaining dye content of 2.59 mg/L and a dye removal percentage of 98.68%. This is because activated charcoal from coffee grounds provides many wide pores that can interact and adsorb naphthol dyes, so with a small dose of adsorbent, it can adsorb naphthol dyes maximally. The greater the number of adsorbent doses added, the greater the naphthol dye absorbed. This phenomenon occurs because the number of particles and adsorbent surface area increases

proportionally so that the active side of adsorption and adsorption efficiency also increase [13]

Increasing the adsorbent dose to 0.750 and 1 gram resulted in an increase in the remaining color content to 5.07 mg/L and 6.22 mg/L and also the percentage of dye removal decreased to 97.41% and 96.82%. This is because the adsorbent used has passed the saturation point so increasing the adsorbent dose can reduce the adsorption process occurred and does not have a significant effect [2].

3.7 Effect of Stirring Time on Adsorption of Naphthol Dye Levels

The stirring time of the adsorbent in liquid waste from the weaving industry can influence the level of adsorption results. Determining the appropriate stirring time can be done by considering the characteristics of the adsorbent and the nature of the dye to be adsorbed. The effect of stirring time can be determined by varying the stirring time of activated charcoal from coffee grounds with varying stirring times of 10; 20; 30; 40 and 50 minutes, using the best adsorbent dose obtained from the adsorption process with varying adsorbent doses, namely 0.500 grams. After the treatment of the liquid waste from the weaving industry, the results obtained are shown in **Table 3**.

Table 2 Adsorption results of naphthol dye levels with varying stirring time

Time (minutes)	Remaining dye levels (Ce)	Decrease in dye levels (%)
10	7.3696±0.15373 ^a	96.2283±0.07863 ^e
20	6.1087±0.33814 ^b	96.8736±0.17310 ^d
30	2.5218±0.12297 ^c	98.7094±0.06293 ^c
40	0.7174±0.15373 ^e	99.6329±0.07870 ^a
50	1.5217±0.30745 ^d	99.2212±0.15733 ^b

Note: Homogeneity data is marked with a superscript letter. The letters 'a' to 'e' indicate the highest value to the lowest

Table 3 shows that increasing the stirring time from 10 to 40 minutes significantly (P -value < 0.05) affects the percentage of color removal in weaving industry wastewater. Equilibrium occurred at 40 minutes with the remaining color content of 0.72 mg/L and the color content removal percentage of 99.63%. This is because the longer the contact time of activated charcoal from coffee grounds with wastewater, the greater the level of naphthol dye that is absorbed. This is because the longer the opportunity for activated charcoal particles to interact with the weaving industry wastewater, the more coloring substances are absorbed by the activated charcoal.

Increasing the stirring time to 50 minutes increased the residual color content to 1.52 mg/L and the percentage of dye decreased to 99.22%. This is because the longer stirring time can also result in a desorption process, namely the release of dyes that have been bound to the activated charcoal. After all, the adsorption process has passed the saturation point and is unable to bind the dyes again [5][14]. According to Dwijayanti et al. [15], once adsorption equilibrium occurs, increasing the stirring time does not have a significant impact on the adsorption of the dye.

4. CONCLUSION

Based on research on the reduction of naphthol dye using an adsorbent from activated charcoal made from coffee grounds, the results obtained were that from the characterization of activated charcoal on coffee grounds the water content was 3.74%, the ash content was 2.12%, and the iodine adsorption capacity was 758%. The results of the characterization of coffee grounds activated charcoal meet SNI 06-3730-1995.

The dose of activated charcoal adsorbent from coffee grounds has an effect on reducing the levels of naphthol dye in liquid waste from the weaving industry. The best adsorbent dose in this study was obtained at a dose of 0.500 grams with an efficiency of reducing naphthol dye levels in weaving industry wastewater of 98.68%.

The time of stirring activated charcoal from coffee grounds affects reducing the levels of naphthol dye in liquid waste from the weaving industry. The best stirring time in this study was found to be 40 minutes with an efficiency of reducing naphthol dye levels in weaving industry wastewater of 99.63%.

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CONFLICT OF INTEREST

No potential conflict of interest was reported by the author(s).

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

Adlina Fitry: conceptualization, investigation, writing—original draft preparation.

Neni Damajanti: conceptualization, methodology, writing—review and editing, supervision.

Yeti Rusmiati Hasanah: data analysis, writing—review, and editing.

All authors have read and agreed to the published version of the manuscript.

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