

## SYNTHESIS OF ACTIVATED CARBON FROM STALKS WASTE OF SORGHUM (*Sorghum bicolor* (L.) Moench) AS A RHODAMINE B ADSORBENT WITH VARYING NaOH ACTIVATOR CONCENTRATIONS

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### ABSTRACT

Rhodamine B, a common synthetic dye used in the textile industry, poses an environmental risk. Its excessive release can alter pH of water, disrupting aquatic microorganisms and animals. Utilizing activated carbon derived from stalks waste of sorghum presents a potential solution for Rhodamine B pollution. Activated carbon's extensive pore structure, developed through activation, provides a large surface area that enhances its adsorption capacity. This study aimed to characterize activated carbon produced from stalks waste of sorghum and determine the optimal NaOH concentration for its synthesis as an adsorbent of Rhodamine B dye. The experimental stages involved preparing stalks waste of sorghum samples, carbonization, and NaOH activation (2, 3, 4, 5, and 6% concentrations for 24 hours). The resulting activated carbon was then used to adsorb Rhodamine B dye. Characterization of the activated carbon revealed a moisture content of 4-7.3%, an ash content of 2.3-7.6%, and an iodine adsorption capacity of 2,410.67-3,427.11 mg/g, met the Indonesian National Standard (SNI). The optimum NaOH activator concentration for Rhodamine B adsorption was 6%, achieving an adsorption efficiency of 88.94%.

**Keywords:** *Activated Carbon, Rhodamine B, Adsorption, Sorghum Stalks, NaOH Activator.*

### INTRODUCTION

Industrial development, concurrent with technological advancements, can generate toxic waste capable of impacting human life (Hardiani and Khair, 2022). This is attributable to inadequate waste treatment practices within the textile industry. Textile industrial waste commonly contain non-biodegradable compounds, which can lead to environmental pollution, particularly in aquatic ecosystems (Musafira *et al.*, 2019). Dye waste represents a category of waste that can impair environmental aesthetics and exhibits toxicity towards both humans and animals. Dye waste can impede sunlight penetration into underwater systems, consequently disrupting biological processes within the aquatic environment (Aisah *et al.*, 2018).

Rhodamine B is a commonly used synthetic dye, particularly in the textile industry. It is an odorless, green crystalline powder that exhibits a red-violet hue at high concentrations and appears bright red at lower concentrations. Excessive levels of Rhodamine B in the environment can cause pH alterations in aquatic systems, disrupting the balance of living organisms and microorganisms. In humans, accumulation of this dye may lead to serious health problems, including hepatic carcinoma, poisoning, respiratory tract irritation, skin irritation, and gastrointestinal disturbances.

Adsorption is considered the most effective method for dye removal due to its safety profile, absence of detrimental health side effects, requirement for simple and inexpensive equipment, ease of implementation, efficiency, and cost-effectiveness. It is a process involving the accumulation of an adsorbate onto the surface of an adsorbent, driven by attractive forces between the adsorbate molecules and the adsorbent surface. An adsorbent is a solid material capable of sorption, whereas an adsorbate can be a solid, liquid, or gaseous substance that is sorbed (Lano *et al.*, 2020). Adsorbents typically utilize porous natural materials with surface areas ranging from 100 to 300 m<sup>2</sup>/g, where the adsorbent surface area is crucial for the

adsorption process. Commonly employed adsorbents include activated carbon, zeolite, and silica gel (Amanda *et al.*, 2022).

Sorghum (*Sorghum bicolor* (L.) Moench) is a cereal plant cultivated by Indonesian farmers, although the land area is relatively small. Traditionally, sorghum is widely planted in East Nusa Tenggara Province, West Nusa Tenggara Province and Central Java Province (Irawan and Sutrisna, 2011). Sorghum cultivation has traditionally focused on its seeds as a rice substitute, leading to the disposal of the stalks as waste. However, these stalks contain significant chemical components—45% cellulose, 27% hemicellulose, and 21% lignin—rendering them a potential source for activated carbon production (Kim and Day, 2011).

This study aims to identify the characteristics of activated carbon produced from sorghum stalk waste and to determine the optimum NaOH concentration for its synthesis as an adsorbent for Rhodamine B dye. Previous research has investigated the use of activated carbon from sorghum stalk waste as an adsorbent, utilizing 40-mesh sorghum bagasse modified with NaOH for the adsorption of Pb(II) heavy metal ions. The findings indicated that optimal adsorption conditions were achieved at a NaOH concentration of 0.04 M, a contact time of 15 minutes, an adsorbent dose of 6 g/L, and an initial Pb concentration of 80 mg/L, with a pH of 5 and a temperature of 35°C. Under these conditions, the adsorption capacity reached 8.1530 mg/L, with a removal efficiency of 60.1408%.

## RESEARCH METHOD

In this study, we utilized sorghum stalk samples from Sainoni Village in North Central Timor Regency, along with NaOH (Merck), Iodine (Merck), KI (Merck), Sodium Thiosulfate (Merck), amylum/starch (Merck), and Rhodamine B (Merck).

### Sample Preparation

The sorghum stalks used are 60 days old (days after planting), with the middle section cut into small pieces measuring 0.5 cm. These are washed thoroughly with water, dried in the sun for two days, and then dried again in an oven at 105 °C until a constant weight was achieved (Batu *et al.*, 2022).

### Sample Carbonation

The dried stalks waste of sorghum was carbonized in a furnace at 400°C for 2 hours. Subsequently, we cooled the resulting carbon in a desiccator, ground it using a mortar and pestle, and sieved it through an 80-mesh to obtain a uniform particle size (Lb *et al.*, 2023).

### Carbon Activation of Sorghum Stalks

Sorghum stalks carbon weighing 20 grams were immersed in 200 mL of NaOH solutions with varying concentrations of 2, 3, 4, 5, and 6%. The mixtures were stirred for approximately 5 minutes and then left to stand for 24 hours. Following this process, the solid was separated by filtration and rinsed with distilled water until a neutral pH was attained. The obtained residue was then dried in an oven at a temperature of 105°C for 3 hours after which it was weighed and analyzed for water content, ash content, iodine adsorption capacity, and its functional groups using a Fourier Transform Infrared (FTIR) Spectrophotometer (Budiman *et al.*, 2018).

### Characterization of Activated Carbon

#### Moisture Content

A mass of 1 gram of activated carbon was weighed using a previously dried porcelain crucible. Subsequently, the sample was placed in an oven maintained at a temperature of 105°C for a duration of 3 hours. Following the heating process, the activated carbon sample was cooled in a desiccator prior to reweighing (Krismayanti *et al.*, 2019). We calculated the moisture content using the formula below.

$$\text{Moisture Content (\%)} = \frac{(a-b)}{a} \times 100\% \dots\dots\dots (1)$$

Notation:

a : initial weight (g)

b : final weight (g)

### Ash Content

A mass of 1 gram of activated carbon was weighed using a porcelain crucible of known weight and subsequently placed in a furnace at a temperature of 800°C for a duration of 2 hours. Following this, the sample was cooled in a desiccator and weighed until a constant weight was achieved (Batu *et al.*, 2023). The ash content can be calculated using the following equation.

$$\text{Ash Content (\%)} = \frac{\text{sample weight} - \text{ash weight}}{\text{sample weight}} \times 100\% \dots\dots\dots (2)$$

### Iodine Adsorption Capacity

A mass of 1 gram of activated carbon was weighed and mixed with 50 mL of a 0.1 N iodine solution. This mixture was then stirred for 15 minutes. Upon completion of the shaking process, the solution was filtered, and 10 mL of the filtrate was collected. This filtrate was subsequently titrated using a 0.1 N sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) solution. As the yellow color began to fade, a 1% starch solution was added as an indicator, causing the solution to turn dark blue. The titration process was continued until the blue color disappeared, resulting in a clear solution (Huda *et al.*, 2020).

$$\text{Iodine Adsorption Capacity} = \frac{A - \left( \frac{B \times N (\text{Na}_2\text{S}_2\text{O}_3)}{N (\text{Iodine})} \right) \times 126.93 Fp}{a} \dots\dots\dots (3)$$

Notation:

A : volume of iodine solution (mL)

B : volume of used Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (mL)

fp : dilution factor

a : weight of activated carbon (g)

N (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) : concentration of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (N)

N (Iodin) : concentration of Iodine (N)

126.93 : total Iodine in proportion to 1 mL of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution

### Application of NaOH-Activated Carbon as an Adsorbent for Rhodamine B Dye

A mass of 1 gram of activated carbon was weighed and added to 50 mL of a 30 ppm Rhodamine B dye solution. Subsequently, the solution was agitated using a shaker for 30 minutes at a speed of 200 rpm. Following this, the solution was filtered using filter paper, and the concentration of Rhodamine B was measured using a UV-Vis Spectrophotometer (Mading *et al.*, 2024). The adsorption capacity can be calculated using the following **Formula 4** (Anggriani *et al.*, 2021):

$$\text{Adsorption capacity} = (q) \frac{C_o - C_e}{m} \times V \dots\dots\dots (4)$$

Notation:

q : adsorption capacity (mg/g)

Co : initial concentration of Rhodamine B (mg/L)

Ce : final concentration of Rhodamine B (mg/L)

V : volume of Rhodamine B solution (mL)

m : mass of activated carbon used (g)

## RESULTS AND DISCUSSION

### Carbonation of Sorghum Stalks Waste

Carbonization is a process involving the volatilization of water and the decomposition of components present in stalks of sorghum, such as hemicellulose, cellulose, and lignin (Handayani and Suryaningsih, 2019). In this study, the carbonization process was conducted at a temperature of 400°C, which is considered an ideal temperature for the carbonization of the sample. At this temperature, the decomposition of lignocellulosic content within the stalks waste of sorghum into carbon occurs (Anggraeni *et al.*, 2023). The decomposition of hemicellulose took place within a temperature range of 200°C to 250°C, indicated by the appearance of thin, white smoke from the furnace chimney. Meanwhile, cellulose decomposition commenced at 280°C and continued until reaching a temperature between 300°C and 350°C, during which thicker and darker smoke, specifically brownish-black, was observed. Subsequently, lignin decomposition initiated at temperatures between 300°C and 350°C and concluded at approximately 400 °C. At this stage, the generated smoke was again white and thin, gradually dissipating (Maruf and Saputri, 2023). The resulting carbon was then ground and sieved using an 80 mesh sieve to reduce particle size and consequently increase the surface area (Siregar *et al.*, 2015). The carbon formed from the carbonization process can be seen in **Figure 1** below.



**Figure 1. Carbon of sorghum stalks waste**

### Activation of Sorghum Stalks Waste Carbon

Activation is a process employed to render carbon active by oxidizing hydrocarbon molecules (Saragih *et al.*, 2020). This process aims to expand the pore surface area of carbon that is obstructed by residual combustion products generated during the carbonization process, thereby enhancing the adsorption capacity of the activated carbon (Lubis *et al.*, 2020). In this study, the activation method utilized was chemical activation, as it offers the advantage of producing larger pores in the activated carbon, which can increase its surface area with the aid of an activating agent (Fadlilah *et al.*, 2022). The activating agent used in this study was sodium hydroxide (NaOH) with varying concentrations of 2, 3, 4, 5, and 6%. The purpose of employing different concentrations of the NaOH activator was to analyze its effect on the characteristics and adsorption capacity of the resulting activated carbon (Winoto *et al.*, 2020).

The activation of carbon with NaOH solution leads to an increase in the size of the carbon pores. This is attributed to the hygroscopic nature of NaOH, which can remove impurities present in the carbon, consequently increasing the number of carbon pores. During the activation process of carbon derived from stalks waste of sorghum, NaOH will be adsorbed, leading to the breaking of bonds in inorganic compounds. Subsequently, these inorganic minerals will dissolve, resulting in the opening of the pores of the activated carbon (Nurhasanah *et al.*, 2024).

### Characterization of Activated Carbon from Sorghum Stalks

The characterization of the activated carbon in this study was conducted to determine its quality by varying the NaOH activator in accordance with the stipulations of the Indonesian National Standard SNI 06-3730-1995 (Siahaan *et al.*, 2013). The characterization performed in this study encompassed the measurement of moisture content, ash content, and iodine adsorption. The characterization results of both non-activated carbon and NaOH-activated carbon derived from stalks waste of sorghum are presented in **Table 1**.

**Table 1. Results of determining moisture content, ash content and iodine adsorption capacity of carbon without-activation and activated-by-NaOH from stalks waste of sorghum**

Type of Carbon	Concentration of NaOH (%)	Moisture content (%)	Ash content (%)	Iodine adsorption capacity (mg/g)
Without activator	0	9.3	18.3	2,030.88
NaOH Activator	2	7.3	7.6	2,410.67
	3	5.6	6.3	2,813.615
	4	5.3	5.6	2,855.925
	5	4.3	4.6	2,898.235
	6	4.0	2.3	3,427.11
SNI 06-3730-1995		≤ 15	≤ 10	> 750

### Moisture Content

Moisture content is a key parameter that can influence the quality of activated carbon. The moisture content test was conducted to determine the hygroscopic nature of the carbon and to quantify the amount of water present within the activated carbon (Nafi'ah, 2016). The evaporation of water molecules from the activated carbon leads to an expansion of its surface area, attributed to the increased porosity of the carbon (Aulia and Khair, 2022). Based on the results presented in **Table 1** above, an increase in NaOH concentration correlates with a decrease in the moisture content of the activated carbon. This is because a higher NaOH concentration employed during activation enhances the ability of NaOH to absorb water, resulting in a lower moisture content value (Fatimura *et al.*, 2020). The reduction in moisture content is influenced by the NaOH activator, which possesses moisture-absorbing properties. Water molecules present within the activated carbon are bound by the NaOH activating agent, leading to an increase in the pores and surface area of the activated carbon; consequently, the adsorption capacity of the activated carbon increases significantly (Batu *et al.*, 2023). The moisture content of the activated carbon across all tested concentrations met the quality standards for activated carbon according to SNI No. 06-3730-1995, which specifies a maximum value of ≤15%.

### Ash Content

The ash content test aims to determine the level of inorganic residue present in the activated carbon. This is crucial because a higher ash content generally correlates with a reduced adsorption capacity. Therefore, efforts are made to minimize the ash content to ensure the adsorption process occurs optimally (Nurrahman *et al.*, 2021). The results presented in **Table 1** indicate that increasing the NaOH concentration during the activation process leads to a decrease in the ash content of the resulting activated carbon. A lower ash content in activated carbon signifies higher quality (Hendrawan *et al.*, 2017). The lower ash content observed in the activated carbon is attributed to the ability of the NaOH activating agent to dissolve impurities in the form of metal oxides and residual inorganic minerals that may block the pores of the activated carbon during the activation process. Most of these residual minerals in the activated carbon are removed during activation, thus no longer obstructing the pores of the activated carbon (Sitanggang *et al.*, 2017). The ash content testing of the activated carbon samples met the quality standards of SNI 06-3730-1995, whereas the non-activated carbon (0% NaOH) did not meet the standard as it exceeded the established maximum ash content of 10%.

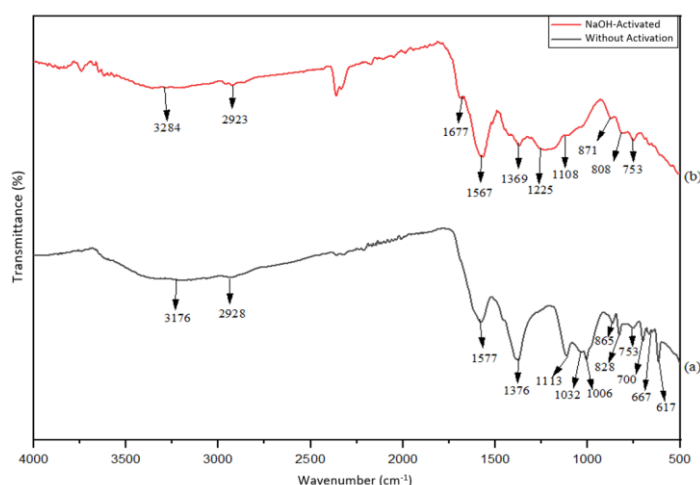
### Iodine Adsorption Capacity

The iodine adsorption capacity test aims to quantify the extent to which activated carbon can adsorb various impurities and dyes present in a solution (Maulana *et al.*, 2017). Based on the results in **Table 1**, an increase in the NaOH concentration employed during the activation process leads to an enhancement in the iodine adsorption capacity of the activated carbon. This is attributed to the fact that a higher concentration of the NaOH activator causes the pores within the activated carbon to become more open, consequently resulting in a greater adsorption capacity (Nurrahman *et al.*, 2021). The increased iodine adsorption is due to the removal of impurities from the surface of the activated carbon pores, which leads to the activation of new pores previously blocked by these impurities, as well as the expansion of the activated carbon's surface area. The relatively high iodine adsorption values obtained indicate that the activated carbon possesses a considerably good adsorption capability (Ilimu *et al.*, 2023). The iodine adsorption testing of the activated carbon samples complied with the specifications of the SNI 06-3730-1995 standard, which requires a minimum of 750 mg/g. Thus, the activated carbon produced from stalks waste of sorghum can be effectively utilized as an adsorbent.

### Functional Groups of Activated Carbon from Sorghum Stalks

FTIR analysis was conducted to identify the functional groups present in the stalks waste of sorghum carbon before and after activation. The resulting FTIR spectra are presented in **Figure 2**.

In **Figures 2a** and **2b**, the FTIR spectra reveal differences in functional groups between the spectrum of non-activated sorghum stalks waste carbon and that of activated carbon. The spectrum of non-activated sorghum stalks waste carbon (**Figure 2a**) exhibits an absorption band at a wavenumber of  $2928\text{ cm}^{-1}$ , indicating the presence of C-H alkane functional groups, while the broad peak at  $3176\text{ cm}^{-1}$  suggests the presence of O-H functional groups from carboxylic acids. The absorption band at a wavenumber of  $1577\text{ cm}^{-1}$  indicates the presence of aromatic C=C functional groups, and the wavenumber of  $1376\text{ cm}^{-1}$  suggests the presence of C-H alkanes, while the wave number of  $1113\text{ cm}^{-1}$  indicates C-O groups. The absorption band observed at a wavenumber of  $865\text{ cm}^{-1}$  indicates the presence of aromatic C-H functional groups (Irnamera, 2020). **Figure 2b** displays the FTIR spectrum of NaOH-activated carbon, showing a shift in the C=C group from a wavenumber of  $1577\text{ cm}^{-1}$  to  $1677\text{ cm}^{-1}$ . The C-H functional group also shifted from a wavenumber of  $1376$  to  $1369\text{ cm}^{-1}$ . These shifts indicate alterations in the functional groups and the emergence of new functional groups during the activation process using NaOH, as evidenced by the appearance of an absorption peak at a wavenumber of  $1677\text{ cm}^{-1}$ , which indicates C=O groups for aldehydes (Astuti *et al.*, 2016).



**Figure 2. Showing the FTIR spectrum of sorghum stalks waste: (a) carbon without activator; and (b) NaOH-activated carbon**



The results of the functional group identification of both carbon and NaOH-activated carbon using FTIR spectroscopy demonstrate the presence of O-H, C-H, C=C, C=O, and C-O functional groups. The presence of O-H, C=O, and C-O bonds suggests that the resulting activated carbon tends to possess more polar characteristics. Consequently, this activated carbon can be effectively utilized as an adsorbent (Nafi'ah, 2016).

### Results of Rhodamine B Adsorption Using Activated Carbon from Stalks Waste of Sorghum

The results of Rhodamine B adsorption using activated carbon produced from stalks waste of sorghum with various concentrations of NaOH activator are presented in **Table 2**.

**Tabel 2. The results of Rhodamine B adsorption using activated carbon from stalks waste of sorghum**

Concentration of NaOH (%)	C <sub>o</sub> (ppm)	C <sub>e</sub> (ppm)	E%
2	30	4.516	84.96
3		3.995	86.81
4		3.828	87.24
5		3.458	88.47
6		3.317	88.94

Based on the study findings presented in **Table 2**, there is an increase in the adsorption capacity of activated carbon derived from stalks waste of sorghum towards the Rhodamine B dye. This is attributed to the fact that as the NaOH concentration used during activation increases, the adsorption efficiency for the Rhodamine B dye also increases (Erlina *et al.*, 2015). The increase in NaOH activator concentration is directly proportional to the increase in the number of pore distribution and the surface area of the activated carbon. Consequently, the number of adsorption sites for the Rhodamine B dye increases, leading to a corresponding increase in efficiency. The larger pore sizes in the activated carbon derived from stalks waste of sorghum can lead to a greater exposure of active sites on the surface of the stalks waste of sorghum, thereby enhancing the adsorption capacity (Nurfitria *et al.*, 2019). According to the research results, the maximum efficiency of NaOH activator concentration in the process of producing activated carbon from stalks waste of sorghum for the adsorption of Rhodamine B dye was achieved at a 6% NaOH concentration, with an adsorption efficiency reaching 88.94%.

### CONCLUSION

Based on the study findings, the following are our conclusions.

1. The characteristics of the NaOH-activated carbon of sorghum stalks waste, based on the results of this study, indicate a moisture content ranging from 4.0% to 7.3%, an ash content ranging from 2.3% to 7.6%, and an iodine adsorption capacity ranging from 2410.67 mg/g to 3427.11 mg/g. Consequently, the NaOH-activated carbon derived from stalks waste of sorghum meets the Indonesian National Standard (SNI) 06-3730-1995. The functional groups identified in the carbon produced from stalks waste of sorghum include O-H, C-H alkane, C-H aromatic ring, C-O, C=O, and C=C. The presence of O-H, C=O, and C-O bonds suggests that the resulting activated carbon tends to exhibit more polar characteristics, thus enabling its utilization as an adsorbent.
2. The optimum concentration of the NaOH activator in the process of producing activated carbon from stalks waste of sorghum, as determined by testing its adsorption capacity for Rhodamine B dye, was found to be 6%, yielding an adsorption efficiency of 88.94%.

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