BIS ENERGY AND ENGINEERING

Volume 3 2026 | DOI: 10.31603/biseeng.595
7th Borobudur International Symposium on Science and Technology (BIS-STE) 2025

UNIMMA PRESS
Universitas Muhammadiyah Magelang

Utilization of ironwood sawdust waste as an adsorbent through chemical and physical activation processes with various KOH activation concentrations

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Abstract

Indonesia's total sawnwood production reaches 2.6 million m^3 per year with the amount of sawmill waste produced amounting to 1.4 million m^3 per year. In ironwood sawdust there is a content of cellulose 39-45%, hemicellulose 15-25%, lignin 18-33%. This study aims to determine the effect of KOH activator concentration on the quality of activated charcoal from ironwood sawdust so that it complies with the Indonesian National Standard (SNI 06-3730-1995). The process of making activated charcoal from ironwood sawdust through a carbonization process using a furnace at a temperature of 500°C for 1 hour, then the charcoal is ground and screened with a size of -80 + 100 mesh followed by chemical activation using KOH activator 3%, 5%, 10%, 15% and 20% for 24 hours and continued with physical activation at a temperature of 700°C for 2 hours. The quality of activated charcoal from ironwood sawdust was tested based on (SNI 06-3730-1995) including water content, ash content, volatile matter content and iodine absorption capacity. The results showed that the best condition of activated charcoal from ironwood sawdust was produced in a physical activation process with a KOH concentration of 10% which resulted in a water content of 1.2808%, ash content of 4.1017%, volatile matter content of 7.9887% and iodine absorption capacity of 812.0556 mg/g.

Published:
May 04, 2026

Selection and Peer-review under the responsibility of the 7th BIS-STE 2025 Committee

Keywords

Activation, Activated charcoal, KOH, Ironwood sawdust

Introduction

Assuming that the waste generated is 54.24% of the total, Indonesia's annual sawn timber production of 2.6 million m^3 results in the production of 1.4 million m^3 of wood sawing waste annually. The Forest Products Research Institute at sawmills in Kalimantan shows that the average sawing yield is 45%, with the remaining 55% being waste. 10% of

the sawmill waste is sawdust. As wood production increases annually, the amount of sawdust waste generated will also increase [1].

Ulin wood sawdust contains important chemical components, such as cellulose (60%), lignin (18-33%), and pentosane (12.7%) [2]. Due to its high cellulose content, sawdust has great potential as a raw material for activated charcoal. Cellulose, with its bound OH groups, will release hydrogen and oxygen atoms during high-temperature carbonization [3], leaving behind carbon atoms that create a porous structure in activated charcoal, thus enabling it to function as an adsorbent.

Physical activation and chemical activation are the two main methods for activating activated carbon. Chemical activation using potassium hydroxide (KOH) is known to expand the formation of micropores and mesopores, which increases the specific surface area of activated carbon. It is suspected that the characteristics and effectiveness of the resulting adsorbent are influenced by fluctuations in KOH concentration.

Activated carbon is typically produced through carbonization followed by an activation process. The activation used is chemical activation with KOH as the activator. KOH is employed because it reacts with the charcoal and removes impurities, thereby enhancing its porosity and adsorption capacity [4].

According to the research by [5], it was proven that using KOH activator resulted in better activated charcoal quality compared to using H_3PO_4 activator. Therefore, this study was conducted using variations in KOH concentration on the quality of wood sawdust activated charcoal as an adsorbent, referring to the standard quality values for activated charcoal based on SNI 06-3730-1995.

Most studies have shown that chemical and physical carbonization and activation can be used to produce activated carbon from various biomass, such as fruit peels, coconut shells, corncobs, sawdust, and other agricultural wastes. Chemical activators such as KOH, $ZnCl_2$, and H_3PO_4 are commonly used because they have been shown to significantly increase the specific surface area and pore volume of activated carbon.

Several previous studies have reported the successful use of various biomass as raw materials for activated carbon, such as coconut shells, sawdust, fruit peels, corncobs, and other agricultural waste through carbonization and chemical and physical activation processes [6], [7]. Commonly used chemical activators include KOH, $ZnCl_2$, and H_3PO_4, which have been shown to significantly increase the specific surface area and pore volume of activated carbon [8], [9].

Several studies have shown that activation using KOH results in a more developed micropore structure and a higher adsorption capacity for dyes and heavy metals due to more optimal pore structure development compared to conventional physical activation [10]. Furthermore, variations in activator concentration and activation

temperature have been reported to directly influence the physicochemical characteristics and adsorption efficiency of the resulting activated carbon [11].

However, specific studies on the utilization of ironwood waste (ulin) with variations in KOH concentration and simultaneous comparison between chemical and physical activation are still limited, so this research is important to be carried out to fill this gap.

From a technological perspective, numerous reports have been made on the combination of physical and chemical activation using KOH with lignocellulosic biomass [12]. Therefore, the innovation of this study does not lie in the technological method; instead, this study focuses on the selection of a specific raw material, namely ironwood powder, and the systematic evaluation of the impact of varying KOH concentrations from 3 to 20%, directly linked to the quality parameters stipulated by the national standard. This study is the first to systematically evaluate the impact of KOH concentration on powder-based activated carbon. Thus, to meet the national standard, this study provides specific experimental data on ironwood material that has not been thoroughly documented before.

Although many biomasses have been studied as raw materials for activated carbon, studies specifically using ironwood waste powder as a precursor with controlled variations in KOH concentration are still very limited. This is especially true for the low to medium concentration range (3–20%) combined with direct comparison to physical activation at the same high temperature conditions. Not many studies have systematically analyzed the impact of variations in KOH concentrations of 3%, 5%, 10%, 15%, and 20% on the characteristics and adsorption capacity of ironwood powder activated carbon.

Therefore, the purpose of this study was to see how changes in KOH concentrations of 3%, 5%, 10%, 15%, and 20% affect the characteristics and adsorption capacity of ironwood powder activated carbon. The hypothesis of this study is that increasing the KOH concentration to a certain limit will increase pore growth and adsorption capacity, but too high a concentration will cause damage to the carbon structure, which in turn will reduce the adsorbent performance.

Method

Activated carbon characterization was performed using laboratory equipment. The carbonization and activation processes were carried out using a muffle oven with digital temperature control up to 1000°C. A digital laboratory oven was also used to dry the samples up to 250°C, and weighing was carried out using an analytical balance with an accuracy of 0.0001 g. Analysis of the methylene blue solution concentration was carried out using a UV-Vis spectrophotometer with a maximum wavelength of 664 nm.

For proximate analysis, national and international standards for moisture and ash content were used. Moisture content was determined based on SNI 06-3730-1995 on technical activated carbon or the equivalent method ASTM D2867 (standard test

method for moisture content in activated carbon). Ash content was determined based on ASTM D2866 (standard test method for total ash content of activated carbon).

Iodine value was calculated using the iodometric titration method regulated by ASTM D4607 (Standard Test Method for Determining the Iodine Value of Activated Carbon). The process involved adsorbing a standard iodine solution on the activated carbon, filtering it, and titrating the filtrate using sodium thiosulfate ($Na_2S_2O_3$) solution with a starch indicator until the endpoint was reached. The methylene blue adsorption test used a UV-Vis spectrophotometric method commonly used to characterize biomass-based activated carbon. Adsorption capacity (mg/g) was calculated by measuring the initial and final concentrations.

The research process begins with the preparation of raw materials, specifically ulin wood sawdust sourced from a ulin wood sawmill. The pre- treatment process for the sawdust involves reducing its size and thoroughly cleaning it. Following this, the sawdust is dried in the sun for three days.

The activator solution was prepared by weighing 3 grams of potassium hydroxide (KOH) to achieve a concentration of 3%. The weighed KOH was dissolved in a 100 ml beaker, transferred to a 100 ml volumetric flask, and distilled water was added until the solution reached the mark. The mixture was then homogenized. This procedure is repeated to prepare KOH solutions with concentrations varying from 3%, 5%, 10%, 15%, and 20%.

The process of making charcoal from wood sawdust begins with carbonizing ironwood sawdust. The raw material was placed in a furnace and heated at 500°C for 1 hour. After the carbonization process is complete, the charcoal is removed and cooled in a desiccator for approximately 15 minutes. Afterward, the charcoal was ground and sieved using an -80 mesh + 100 mesh screen to obtain a uniform particle size. The sieved charcoal was then soaked for 24 hours. Subsequently, an analysis was conducted for moisture content, ash content, volatile matter content, and iodine absorption capacity.

The production of activated charcoal begins with the chemical activation stage. A 14-gram sample of the sieved ulin wood sawdust charcoal with a mesh size of -80 + 100 was weighed. The charcoal was then soaked for 24 hours using 70 ml of KOH activator solution with varying concentrations of 3%, 5%, 10%, 15%, and 20%. After soaking, the activated charcoal was filtered from the activator solution using Whatman No. 42 filter paper and washed with distilled water until a neutral pH was reached. The neutralized charcoal was then dried in an oven at 110°C for 3 hours, followed by cooling in a desiccator for 15 minutes. At this point, an initial quality analysis was performed, including moisture content, ash content, volatile matter content, and iodine absorption capacity.

The process continues with the physical activation stage. The dried activated carbon was put back into the furnace and heated at 700°C for 2 hours. Finally, the activated carbon was cooled in a desiccator for 30 minutes, and a final quality analysis was

performed again (moisture content, ash content, volatile matter content, and iodine adsorption capacity).

The moisture content (%) was calculated using equation (1).

$$\text{Moisture Content (\%)} = \frac{(m2-m3)}{(m2-m1)}\ x\ 100\ \% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Notes:
m1 = mass of empty petri dish (g)
m2 = mass of sample and dish before heating (g)
m3 = mass of sample and dish after cooling (g)

The ash content (%) was calculated using equation (2).

$$\text{Ash Content (\%)} = \frac{(m3-m1)}{(m2-m1)} x\ 100\ \% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

Notes:
m1 = mass of empty petri dish (g)
m2 = mass of sample and dish before heating (g)
m3 = mass of sample and dish after cooling (g)

The volatile matter content (%) was calculated using equation (3).

$$Volatile\ Matter\ Content\ (\%) = \frac{(m2-m3)}{(m2-m1)}\ x\ 100\ \% - M \ldots\ldots\ldots(3)$$

Notes:
m1 = mass of empty petri dish (g)
m2 = mass of sample and dish before heating (g)
m3 = mass of sample and dish after cooling (g)
M = moisture content (%)

The adsorption capacity of activated carbon for iodine was analyzed using the iodometric titration method and can be calculated using the following equation (4).

$$\text{Adsorption Capacity I2} = \frac{(\text{Viod x Niod}) - (\text{Vthio x Nthio}) x\ 126.9\ x\ \text{fp}}{\text{W}} \ldots\ldots\ldots\ldots\ldots\ldots(4)$$

Notes :
10 = Sample volume
V_{tio} = Volume of sodium thiosulfate solution required (ml)
N_{tio} = Normality of sodium thiosulfate solution (N)
N_{iod} = Normality of I_2 solution
126.9 = Atomic weight of iodine
W = Sample mass (g)
Fp = Dilution factor

Results and discussion

The results of the analysis of the characteristics of ironwood sawdust charcoal before undergoing chemical and physical activation with varying concentrations of KOH activator are shown in Table 1.

Table 1. Data from the analysis of charcoal before activation

No.	Parameter	Value
1.	Moisture content (%)	5.23
2.	Ash content (%)	18.87
3.	Volatile matter content (%)	20.85
4.	Adsorption Capacity I_2 (mg/gr)	39.28
5.	Particle size (mesh)	-80 +100

The results of the activated carbon analysis after the activation process refer to the standard quality values of activated carbon based on SNI 06-3730- 1995, as shown in Table 2.

Table 2. Standard quality values of activated carbon (SNI 06-3730-1995)

No.	Parameter	Value
1.	Moisture content (%)	Max.15
2.	Ash content (%)	Max. 10
3.	Volatile matter content (%)	Max. 25
4.	Adsorption Capacity I_2 (mg/gr)	Min. 750

Moisture content analysis determines the percentage of water in activated carbon. The presence of water in activated carbon is related to the charcoal's hygroscopic nature; this extremely hygroscopic feature is what makes activated carbon useful as an adsorbent [13]. The lower the water content, the less water remains to coat the pores of the activated carbon. Table 3 displays the results of the moisture content study performed on activated charcoal following the activation procedure.

Table 3. Data from the analysis of activated carbon moisture content

No.	Concentration of KOH (%)	Moisture content (%)
1.	3	1.55
2.	5	1.30
3.	10	1.28
4.	15	1.27
5.	20	1.26

The results of the moisture content testing in Table 3 show a negative correlation between KOH concentration and moisture content, where increasing the KOH concentration from 3% to 20% progressively decreases the moisture content. The lowest moisture content was obtained at a concentration of 20% (1.26%), and the highest at 3% (1.55%). This decrease is attributed to the hygroscopic nature of KOH, which enhances its ability to bind water molecules from the charcoal. Maximum water content reduction will support the physical activation process by maximizing water evaporation, which contributes to the formation of larger and more numerous pores. The total moisture content of the activated carbon produced in this study met the Indonesian National Standard [14], which sets a maximum limit of 15%.

Ash content analysis aims to identify the metal oxide content of charcoal, which is composed of minerals that do not evaporate during the charring process [15]. A high ash content leads to poorer adsorption capacity; thus, efforts are made to keep the ash content as low as possible so that the adsorption process can be optimized. Table 4 displays the results of an examination of ash content in activated charcoal following the activation procedure.

From Table 4, increasing the concentration of KOH tends to decrease the ash content of activated charcoal. This decrease is observed from a concentration of 3% (4.41%) to the lowest ash content at a concentration of 10% (4.10%). The reduction in ash content is

attributed to the increasing capability of the KOH activator to bind and dissolve metal oxides or minerals present in the activated carbon. However, there were fluctuations, with a slight increase in ash content at 15% (4.39%) and 20% (4.37%). This increase is indicated by the potential for KOH to be adsorbed into the activated carbon and remain after the activation process. Despite fluctuations, the total ash content produced in this study still meets the Indonesian Industrial Standard [14], which sets a maximum limit of 10%.

Table 4. Data from the analysis of activated carbon ash content

No.	Concentration of KOH (%)	Ash content (%)
1.	3	4.41
2.	5	4.12
3.	10	4.10
4.	15	4.39
5.	20	4.37

The purpose of analyzing the volatile matter content is to determine or identify the content of compounds that can vaporize at 950°C in activated carbon. During analysis with heating above 900°C, nitrogen and sulfur will vaporize, and these components are what are called volatile matter [16].

Table 5. Data from the analysis of the volatile matter content of activated carbon

No.	Concentration of KOH (%)	Volatile matter content (%)
1.	3	8.85
2.	5	8.49
3.	10	7.78
4.	15	8.28
5.	20	8.23

From Table 5, it can be seen that increasing the concentration of KOH generally leads to a decrease in the content of volatile matter. This decrease occurred at a concentration of 3% (8.85%), reaching its lowest value at 10% (7.78%). This is due to the function of the activator, which helps open the pores of the activated carbon, allowing the volatile substances trapped inside to be released to the maximum extent. A low volatile matter content is a direct indication of the increased adsorption capacity of activated carbon. However, there is an increase in volatile matter content at concentrations of 15% (8.28%) and 20% (8.23%). This increase is believed to be related to the high ash content at that concentration, which likely caused pore blockage and sub optimally hindered the release of fly ash. Nevertheless, the total amount of volatile matter produced in this study met the requirements of the Indonesian Industrial Standard (SNI, 1995), which sets a maximum limit of 25%.

Iodine adsorption analysis aims to determine the adsorption capacity of activated carbon. One method used in analyzing the adsorption capacity of activated carbon toward iodine solutions is the iodometric titration method. This adsorption capacity can be demonstrated by the magnitude of the iodine number, which indicates how much

the adsorbent can adsorb iodine. The higher the iodine number, the greater the adsorption capacity of the adsorbent [17].

Table 6. Data from the analysis iodine adsorption capacity of activated carbon

No.	Concentration of KOH (%)	Iodine adsorption capacity (mg/gr)
1.	3	629.27
2.	5	655.38
3.	10	812.05
4.	15	733.72
5.	20	707.61

The data in Table 6 show a significant positive correlation between KOH concentration and iodine number up to a certain point. Increasing the KOH concentration from 3% to 10% increased iodine adsorption capacity, which reached a maximum value of 812.05 mg/g at a KOH concentration of 10%. This increase indicates that the optimal KOH concentration successfully formed more micropores, thereby enhancing the activated carbon's adsorption ability. However, iodine adsorption capacity then decreased at concentrations of 15% (733.72 mg/g) and 20% (707.61 mg/g). This decrease is caused by the KOH concentration being too high, which triggers the formation of a crust on the surface of the charcoal, leading to pore blockage and reduced access for adsorption. Based on this data, the activated charcoal produced at a concentration of 10% KOH is the only one that meets the requirements of the Indonesian Industrial Standard (SNI-1995), which is a minimum of 750 mg/g.

Conclusion

It was demonstrated that activated carbon produced via chemical-physical activation with 10% KOH at 700^{0}C meets the requirements of SNI No. 06-3730- 1995. Key characteristics include a moisture content of 1.28%, ash content of 4.10%, volatile matter content of 7.78%, and a high iodine adsorption capacity of 812.05 mg/g.

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