Preparation and characterization of a high surface area of activated carbon from *Bambusa vulgaris*—Effect of NaOH activation and pyrolysis temperature

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Abstract. In this study, *Bambusa vulgaris* was used as raw material of activated carbon. The activated carbon was generated using NaOH as an activating agent. The activated carbon produced with the weight ratio of carbon to NaOH 1:1, 1:2, 1:3 and pyrolysis temperature 600, 700 and 800 °C. The experimental result showed that the best characteristic of activated carbon was obtained at weight ratio of carbon to NaOH 1:3 and pyrolysis temperature 800 °C.

1. Introduction

Activated carbon is an adsorbent produced from materials containing carbon [1]. Activated carbon as charcoal was processed using CO₂, water vapor and chemical materials at high temperatures. It can be used as an absorbent [2]. Activated carbon is a porous substance with a high surface area that can absorb and remove pollutants such as heavy metals, dyes, gases and pesticides [3].

In industry, activated carbon is used in purification units, catalysis, recuperation of solvents and hydrocarbon, natural gas and hydrogen storage and energy storage [4]. Furthermore, activated carbon is used in deodorization processes, filtration, discoloration, modification of liquid, gas concentration, petroleum, pharmaceutical, and food and beverage. This absorption capability is caused by its large number of micro pores, which results in capillary symptoms that can absorb pollutants.

Many materials have been used to produce a good quality activated carbon. The effect of chemical substance addition in the preparation of activated carbon was also studied. Chemical substances used in the activation process give many advantages, including single low activation temperatures, activation time, step activation and better porous structure [5]. Activated carbon consists of 87-97% carbon and the rest of the components are oxygen, hydrogen, sulfur, nitrogen, and other compounds formed in the process of producing activated carbon.

Meanwhile, a recent study reported the potency of *Bambusa vulgaris* as a activated carbon source. The activated carbon from yellow bamboo was activated by H₂SO₄ to absorb heavy metals of Cd, Hg and Zn [6]. Yellow bamboo has particularly spread and flourished in tropical Asian regions. The chemical compositions of yellow bamboo were 42.4-53.6% of cellulose, 19.8-26.6% of lignin, 17.5-21.5% of pentose and 1.24-3.77% of ash. Adsorption is the process of transferring adsorbate mass...
from the moving phase into the absorbent surface, which is influenced by the attractive force between adsorbate molecules with active pores on the absorbent surface [2].

In this study, the *Bambusa vulgaris* was used as a source of activated carbon. The activated carbon was chemically activated using NaOH. The characteristics of activated carbon produced will be investigated.

2. Materials and Methods

*Bambusa vulgaris* was obtained from local farmers in Aceh Besar District, Aceh, Indonesia. It is locally known as *Treng Gadeng* by Acehnese society. The chemicals used were HgCl$_2$, HNO$_3$, NaOH, HCl, Aquadest, N$_2$, KMnO$_4$, K$_2$S$_2$O$_8$, NaCl and SnCl$_2$. All chemicals were provided by Merck and used as received.

The carbonization process of *Bambusa vulgaris* was performed with a furnace at temperature of 500 °C and nitrogen gas flows of 200 mL/min for 2 hours. Then the charcoal was cooled and crushed by milling and sieving until -60 +120 mesh of size. After that, *Bambusa vulgaris* charcoal was mixed with NaOH as an activating agent. The weight ratios of carbon to NaOH used were 1:1, 1:2 and 1:3. Then pyrolysis was conducted at temperatures of 600, 700 and 800 °C. Table 1 shows the experimental design. Thereafter, the activated carbon was washed using HCl and stirred for 30 minutes at 85 °C. Finally, residual acid was removed from the product by washing with aquadest.

| Sample code | Weight ratio of carbon to NaOH [g/g] | Pyrolysis temperature [°C] |
|-------------|-------------------------------------|---------------------------|
| AC-1        | without activation                  | 500 °C                    |
| AC-2        | 1:1                                 | 600 °C                    |
| AC-3        | 1:2                                 | 600 °C                    |
| AC-4        | 1:3                                 | 600 °C                    |
| AC-5        | 1:1                                 | 700 °C                    |
| AC-6        | 1:2                                 | 700 °C                    |
| AC-7        | 1:3                                 | 700 °C                    |
| AC-8        | 1:1                                 | 800 °C                    |
| AC-9        | 1:2                                 | 800 °C                    |
| AC-10       | 1:3                                 | 800 °C                    |

The characteristics of the activated carbon include surface area, total pore volume, average pore diameter as determined by the Brunauer, Emmett and Teller (BET) method. It was obtained using Surface Area Analyzer Nova Win Version 11.0 (Quantachrome). The morphology structure of activated carbon was analyzed by means of a Scanning Electron Microscope (TM 3000 Hitachi). All measurements were conducted in the laboratory at ambient temperature; the measurements were collected in duplicate, and the averaged data are reported.

3. Results and Discussion

Figure 1 presents the surface area of activated carbon. The larger the surface area and pore diameter, the better the quality of the activated carbon produced. Figure 1 shows that *Bambusa vulgaris* has 53.5 m$^2$/g of surface area. The pyrolysis temperature has a significant effect for increasing of the surface area of activated carbon. The activated carbon had a surface 308 m$^2$/g when prepared at weight ratio of carbon to NaOH 1:1 and pyrolysis temperature 700 °C. At a similar weight ratio of carbon to NaOH, increasing the pyrolysis temperature up to 800 °C caused a much wider surface area, i.e., 560 m$^2$/g. In addition, the addition of NaOH as the activating agent caused an increase of surface area of activated carbon. The higher the weight ratio of NaOH activator used, the larger the surface area obtained was. At pyrolysis temperature 800 °C, the surface areas of activated carbon at weight ratio of carbon to NaOH 1:1, 1:2 and 1:3 were 560, 635 and 1041,716 m$^2$/g, respectively.
Figure 1. Surface area of the raw material, char, and activated carbon.

Figure 2 presents the total pore volume of activated carbon. There was a similar trend result. The pyrolysis temperature and weight ratio of carbon to NaOH influenced the change in surface area. The total pore volume increased with increasing of pyrolysis temperature and weight ratio of carbon to NaOH.

Figure 2. Total pore volume of raw material, char, and activated carbon.

The pyrolysis temperature and weight ratio of carbon to NaOH caused some changes in the physical properties of activated carbon. The increasing pyrolysis temperature and weight ratio of carbon to NaOH potentially increased the residual loss of hydrocarbons occupying the pore space [7]. The additional activator oxidized and eroded the carbon wall so that it formed more pores and increased the surface area of the carbon, and increasing the heat of chemical activity in the activated carbon also contributed to the production of larger pore diameter [8].

The pore size distribution of activated carbon is a significant characterization in determining the quality product. The activated carbon from rice bran has pore volume with the range of 0.025 to 0.14
cm$^3$/g [9], compared to the total pore volume with the range of 0.15 to 0.622 cm$^3$/g of activated carbon generated from *Bambusa vulgaris* obtained in this study.

Figure 3 shows the average pore diameter of activated carbon, which was in the range of 23.2 to 30.5 Å. The pyrolysis temperature and weight ratio of carbon to NaOH provided specific pore volume. The decline in physical properties might be caused by carbon damage owing to the heating processes during activation. Thus, it can be concluded that the high pyrolysis temperature can influence average pore diameter. The increasing size of pore diameter occurred in several steps, starting with the opening of pores that were previously closed, formation of new pores, and enlargement of the formed pores [3]. The increased pore diameter could also potentially decrease the surface area.

![Figure 3. Average pore diameter of the raw material and char (a), activated carbon (b).](image)

The activated carbon produced in this study was then observed with a Scanning Electron Microscope (SEM) to identify the morphological structure, which is shown in Figure 4. As can be seen in Figure 4, *Bambusa vulgaris* and activated carbon had some pores on their surfaces. The walls of macropores contained many subpores that connected the macropores in the interior of the material.

It can also clearly be seen that a formation of pores and sub-pores rose because of the addition of NaOH as the activating agent. This might be because the activation process with NaOH and the rising pyrolysis temperature increased the reaction between carbon and the activating agent to produce CO$_2$. The reaction is shown Equations (1)-(3). The CO$_2$ produced will diffuse to corrode the carbon wall to form new pores and make pores to be more open [2].

\[
\begin{align*}
4 \text{NaOH} + C & \leftrightarrow 4 \text{Na} + \text{CO}_2 + 2 \text{H}_2\text{O} \quad ............... \quad (1) \\
6 \text{NaOH} + 2 \text{C} & \leftrightarrow 2 \text{Na} + 3 \text{H}_2 + 2 \text{Na}_2\text{CO}_3 \quad ............... \quad (2) \\
4 \text{NaOH} + 2 \text{CO}_2 & \leftrightarrow 2 \text{Na}_2\text{CO}_3 + 2 \text{H}_2\text{O} \quad ............... \quad (3)
\end{align*}
\]

4. Conclusion
The effect of NaOH activation and pyrolysis temperature on characteristics of activated carbon produced from *Bambusa vulgaris* was investigated. The experimental result showed that the best characteristic of activated carbon was obtained at weight ratio of carbon to NaOH 1:3 and pyrolysis temperature 800 °C. At this condition, the surface area, total pore volume and average pore diameter obtained were 1042 m$^2$/g, 0.62 cm$^3$/g and 24 Å, respectively.
Figure 4. Morphological structure of the activated carbon: (a) AC-2; (b) AC-4; (c) AC-7; (d) AC-10.

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