Production and characterization of activated carbon from cashew nut shell using N\(_2\) as activation agent

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Abstract. The activated carbon from cashew nut shell (CNS) has been prepared by physical activation. The cashew nut shell liquid (CNSL) is primarily extracted from a shell by using handed-rotary roaster kiln at the temperature around 170°C. The carbonization is done by augmenting temperature up to 350°C without taking out the CNS from kiln. The carbonized CNS is crushed and sieved of 60 mesh. While the activation process, nitrogen gas is flowed through the electric furnace with a constant flow rate and the activation temperature is varied from 400°C to 900°C. Sample then is characterized using ultimate analysis to determine composition of activated carbon including carbon (C), hydrogen (H), nitrogen (N), oxygen (O) and sulphur (S) and scanning electron microscope (SEM) and Energy Dispersive Spectroscopy (EDS) to know the surface morphology and element composition respectively and X-Ray diffraction (XRD) is used to know the structural properties. The ultimate analysis shows that carbon element in the sample increases as activation temperature increases up to 700°C of 79.80% and then decreases to 79.14% at 900°C. XRD pattern shows that it appears a broaden peak about 2\(\theta\) = 24° indicating an amorphous structure. The increasing of activation temperature tends to graphitization process of activated carbon.

1. Introduction

The research on activated carbon has attached much attention by many researchers because of its variety of applications such as an absorbent, an electric double layers capacitor, a filter, etc. So far, the utilization of biomass from agricultural waste as raw materials on the production of activated carbon has done by several researchers. The agricultural waste such as cashewnut shell [1], rice husk [2], durian shell [3], sugar cane and horn corn [4] have been used to produce activated carbon. National production of raw cashew nut is about 95,000 ton per year and these amounts do not increase significantly among last 10 years. The main producer of raw cashew nut is Southeast Sulawesi (35 % of national production), South Sulawesi (25 %), Lombok, Flores and Sumbawa (30 %) and Jawa-Madura (10 %) [5]. About 35% weight of raw cashew nut consists of CNSL, 25% of nut and the rest is shell. So, it is approximately 38,000 ton CNS available in Southeast Sulawesi in a year. So far, cashew nut shell becomes an agricultural waste in this region. In general, there are two methods to fabricate activated
carbon from agricultural waste such as chemical method and physical method. Chemical activation is done by immersing raw material in the proper chemical solution such as ZnCl₂, H₃PO₄. Physical activation is ideally carried out through 2 (two) steps include carbonization process of raw material and activation process by heating carbonized cashew nut to high temperature. For the physical method, researchers commonly use heat energy such as applied electric furnace or microwave. The use of microwave for material processing recently became popular because of its unique effect on the material such as selective heating and fast heating.

Many researchers have conducted the research on production, and application of activated carbon, but it remains only a few researcher concerns on activated carbon from cashew nutshell. Therefore, this study aims to prepare and to characterize activated carbon from CNS. In our previous work, we prepared activated carbon from CNS only by physical activation without an activation agent [6, 7]. In this study, we prepare the activated carbon from CNS by flowing nitrogen gas into an activated chamber as activating agent while the activation process. Khezami et.al. (2007) reported that the common activation agent is steam, CO₂ and N₂ [8].

2. Methods
The CNS in this research is collected Lombe District in Buton Regency, Southeast Sulawesi. To remove the residual element in raw material, cashew nut shell is washed by aquadest, then it is dried in sunlight exposure. Carbonization process is done through 2 (two) stage. Firstly, the extraction of CNSL by using handheld-rotary coaster kiln [9] in which the temperature is maintained about 170°C. Secondly, without taking CNS out, the heating is continued until the temperature reaches 350°C and it is maintained for 4 hours. Then, carbonized CNS is crushed and sieved in pieces of 60 meshes. The activation process is done by physical activation. Among the activation process, the nitrogen gas is flowed into the electric furnace with a constant flow rate. The activation temperature is varied from 500°C to 900°C with the interval of 150°C and activation time is 30 minutes. Proximate analysis is done to determine moisture content, ash content, volatile matter content and fixed carbon content, then, ultimate analysis is used to determine the element content such as C, O, H and N. ultimate analysis is done in Laboratorium Mineral dan Batubara Bandung and structural properties is characterized by XRD in Laboratorium Kritalografi IPB.

3. Experimental Results and Discussion

3.1. Ultimate analysis and proximate analysis
Table 1 shows the ultimate analysis result of activated carbon from cashew nut shell for different activation temperature. This table provides the information about the effect of temperature on element composition such C, O, H, N and S of activated carbon from CNS. It seems that the C element is a dominant element in the activated carbon from CNS either before or after the activation process. Before activation, C element is about 70.25%. As activation temperature increase, the C becomes 79.80% in activation temperature of 700°C and decreases to 79.14% in activation temperature of 900°C. In relevance, the H and O element decreases as activation temperature increases. This result produced by ultimate analysis shows that the carbon content fulfills the quality standard of activated carbon issued by Standar Nasional Indonesia (SNI) 06-3730-1995 of 65%.

|          | C (%) | H (%) | N (%) | S (%) | O (%) |
|----------|-------|-------|-------|-------|-------|
| carbonized CNS | 70.25 | 4.61  | 0.94  | 0.01  | 20.13 |
| Tₐ = 500°C    | 74.90 | 3.21  | 1.00  | 0.02  | 15.10 |
| Tₐ = 700°C    | 79.80 | 1.93  | 1.03  | 0.01  | 9.22  |
| Tₐ = 900°C    | 79.14 | 1.02  | 1.18  | 0.02  | 8.93  |
Table 2. The proximate analysis result of activated carbon from cashew nut shell for different activation temperature.

|         | Moisture (%) | Ash (%) | Volatile Matter (%) | Fixed Carbon (%) |
|---------|--------------|---------|---------------------|-----------------|
| carbonized CNS | 4.00         | 3.67    | 3.34                | 88.99           |
| $T_a=500^\circ C$ | 1.40     | 1.85    | 1.42                | 95.33           |
| $T_a=700^\circ C$ | 2.40     | 1.04    | 1.02                | 95.54           |
| $T_a=900^\circ C$ | 1.60     | 0.82    | 1.02                | 96.56           |

Proximate analysis provides the information about moisture, ash, volatile matter and fixed carbon in activated carbon. Table 2 shows the proximate analysis result of activated carbon from CNS for different activation temperature. The fixed carbon content of activated carbon is determined by moisture, ash, volatile matter. The results show that the fixed carbon increases regularly as the activation temperature increase. As carbonized, the carbon content is 88.99%. It increases up to 96.56% in activation temperature of 900 °C. In general, the higher activation temperature the higher ash is. In contrast in this study, ash content decreases while activation temperature increases. It indicated that the use of nitrogen as activation agent is effective to prevent the formation of ash from the surface of char.

Figure 1(c-d). SEM micrograph of activated carbon from CNS (a) carbonized CNS (b) activation temperature of 500°C. (c) activation temperature of 700°C and (d) activation temperature of 900°C.
3.2. *SEM and EDX characterization*

The results for scanning electron micrograph (SEM) of activated carbon of CNS for the different activation temperature under 5000 times magnifications are shown in figure 1(a-d). The surface of the carbonized CNS was dense without any cracks and crevices. This surface pattern had poor and negligible adsorption capacity. In contrast, the activated carbon observed in the SEM was found in clear pore structure developed. Many cavities over the surface are well-developed pores. The size of surface pore increases as activation temperature increase.

3.3. *Structural properties by X-ray diffraction*

Figure 2 shows the X-ray diffraction pattern of activated carbon from cashew nutshell. As carbonized, XRD pattern shows a broaden peak located about $2\theta = 24^\circ$ indicating an amorphous structure. The appearance this peak and the peak in $2\theta = 43^\circ$ show the structure similar to graphite. The sharp peak on $2\theta = 38^\circ$ is still unclear. It is convenient to relate to the characteristic of MgO (111) that can be confronted with EDX result. The peak on $2\theta = 43^\circ$ is clearer after activation in which the increasing activation provoked the broader of peak located on $2\theta = 43^\circ$ the appearance two new peaks about $2\theta = 29^\circ$ and $2\theta = 32^\circ$. This result seems to become the starting point toward the occurrence of the graphitization process. Zhao *et. al.* (2009) have reported that based on the evolution of peak intensity of XRD pattern, the graphitization process can be divided into three regions i.e. non-graphitization region, near graphitization region and graphitization region for activation below 900°C, 1000 – 1100°C and over 1200°C respectively [10].

![Figure 2. X-ray diffraction pattern of activated carbon from cashew nut shell with the different activation temperature.](image)

4. *Conclusion*

This study results indicated that cashew nutshell as an inexpensive raw material to produce the activated carbon. Ultimate analysis shows that the activated carbon produced has optimum carbon content on the activation temperature of 700°C. The x-ray diffraction result provided the possibility of graphitization process by increasing the activation temperature.
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