Effect of temperature and time of carbonization on coal-based activated carbon adsorption

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Abstract. Coal from Bangko, South Sumatra is a low rank coal which has the potential to be used as activated carbon material. The result of proximate analysis and calorific value of coal shows that Bangko coal is included in the lignite category, with fixed carbon 43.58%, volatile matter 41.1% and calorific value 4915 Cal/g. Synthesis of activated carbon from lignite coal was carried out through the carbonization and activation. Carbonization was carried out at several temperature variations, between 400◦C - 900◦C for 2 hours using airtight steel box under non-oxidizing conditions. While the activation has taken place at 900◦C for 60, 90 and 120 minutes using water vapour as an activator. The activated carbon iodine number was determined as an indicator of the coal absorption capacity and its porosity. Synthesized activated carbon at 900◦C and 120 minutes shows the highest iodine number, 1274.8 mg / g. This activated carbon has 76.78% fixed carbon, 10.65% volatile matter, 5.91% moisture content and 6.66% ash content. Based on the result, we were concluded that low rank coal like lignite was the potential material to produce coal based activated carbon with high capacity adsorption.

1. Introduction
The world’s coal reserves are dominated by low ranked coal (about 30%), one of them is lignite coal [1]. Lignite coal possesses high level of water rate that result on low heating value, which causes high escalation on fuel consumption and transportation cost [2]. In another word, the high water rate will decrease the coal’s conversion efficiency as the main material for power plants [3]. To improve low rank coal utilization, a clean technology such as liquefaction, gasification, and activated carbon production by utilizing coal as the main material are required.

Activated carbon possesses large surface area and high absorption capacity, so that it can be used as electrode [4], catalyst [5], and adsorbent [6] materials. Activated carbon can be produced from biomasses such as pine wood [7], bamboo [8], coconut skin [9], and low rank coal such as lignite [10] and subbituminous [11]. Activated carbon can also be produced through physics and chemical activations. In physics activation, precursor is activated by using gas such as carbon dioxide and water vapour. Before conducting physics activation, we need to conduct carbonization by heating the sample on a temperature of 600 - 900°C in non-oxidation condition. After that, activation is conducted on of 600 - 1200°C to produce a regular pore structure [12]. In this experiment, activated carbon will be synthesized with lignite coal as precursor through physical activation by water vapour flow.
2. Experimental

2.1. Materials preparation
Coal sample was taken in Bangko, South Sumatera pit 3, on layer C by channel sampling system. Sample preparation was conducted by crushing the sample to produce 60 mesh grain sizes. After that, ultimate and proximate analysis that includes; moisture, volatile matter, ash, fixed carbon, heating value, carbon, hydrogen, nitrogen, and oxygen was conducted.

2.2. Activated carbon synthesize
Activated carbon synthesize was conducted through two stages which are carbonization and activation. Carbonization was done by conducting non-oxygen coal sample heating inside a 3mm thick airtight steel box, fitted with special channel to release flying materials through a fume hood. Carbonization was conducted in several levels of temperature variation between 400°C – 900°C for 2 hours.

Activation was conducted by utilizing water vapour as activation material. Activation was conducted on a temperature of 900°C for 60, 90, and 120 minutes. After that a proximate analysis, yield determination, and iodine number against activated carbon resulted through synthesize process was done. Besides that, iodine number is also determined on standard activated carbon as comparison.

2.3. Iodine number determination
Iodine number measurement was conducted with SII 0258-89 method (SNI 06-3730-1995), scaled as much as 1 gram and placed inside an Erlenmeyer flask, then 10 mL of HCl 5% was added. The solution is further heated until it boils and left for 30 seconds. 100 mL of 0.1 N I₂ was later added then stirred and filtered out. After that, 50mL of filtrate mixed with 1 mL of starch solution was added and titrated with Na₂S₂O₃ 0.1 N solutions. Iodine number is determined with the following formula:

\[
\text{Iodine Number (mg/g)} = \frac{(V1N1 - V2N2) \times 126.9 \times 5}{W}
\]

V1 represents analyzed iodine solution (mL), N1 as iodine normality, V2 as required thiosulfate solution (mL), N2 as sodium thiosulfate normality, and W that represents activated carbon weight.

3. Results and discussion
In this experiment, the utilized coal as activated carbon precursor was analyzed to acknowledge its rank. Based on the proximate, ultimate and calorific value analysis as shown in Table 1, we discover that the utilized coal sample in this research is a low rank lignite coal (based on coal ASTM characteristic).

| Analysis | Analysis Parameter | Result (% adb) |
|----------|-------------------|----------------|
| Proximate | Moisture content | 14.12          |
| | Ash content      | 1.2            |
| | Volatile matter  | 41.10          |
| | Fixed carbon     | 43.58          |
| | Calorific value (Cal/g) | 4915   |
| Ultimate | Sulphur           | 0.4            |
| | Carbon            | 73             |
| | Hydrogen          | 5.9            |
| | Nitrogen          | 0.7            |
| | Oxygen            | 20             |

Synthesis of activated carbon from coal was conducted through carbonization and activation phases. Carbonization was conducted by utilizing an airtight steel box under a non-oxidation condition in several temperature variations. Proximate analysis was conducted towards activated carbon resulted from the synthesize process, shows results as pictured in Figure 1. Based on the figure, it shows that the higher
the carbonization temperature, it results a lower production of moisture content and volatile matter of the coal sample. Meanwhile the fixed carbon will be higher because of the smaller volatile matter production.

Yield is measured based on a comparison between carbonization resulted coal and the original weight of the coal. From table 2, we can see that yield of activated carbon is decreasing in consistent with temperature increase. This situation is produced because high temperature coal will lose more water and volatile matter, which decrease its masses. Besides that, iodine number of activated carbon was also measured. Iodine number is an indicator of activated carbon absorption capacity that shows porosity of activated carbon [13]. Based on the situation, the higher the iodine number, the absorption capacity of the activated carbon is also increased. This is because the increase in temperature causes more moisture and volatile matter to be lost from the coal sample which causes the levels of impurities that block the coal pores decreases [14]. The impurities decrease will shape micro pores and produces a larger surface area [15]. Based on the data obtained through carbonization process, we acknowledge that maximum iodine number is produced through carbonization process on temperature of 900°C, which means that this level of temperature can be utilized in activation process.

Table 2. Yield and iodine number in several carbonization temperature variations.

| Parameter          | 400°C | 500°C | 600°C | 700°C | 800°C | 900°C |
|--------------------|-------|-------|-------|-------|-------|-------|
| Yield%             | 74.5  | 67.25 | 70.5  | 71.25 | 68.75 | 63    |
| Iodine Number, mg/g| 449.5 | 499.6 | 549.6 | 704.7 | 974.9 | 1150.1 |

Activation process conducted in temperature of 900°C by flowing water vapour for 60, 90, and 120 minutes. The activation results are further analyzed to measure moisture, ash, volatile matter and fixed carbon, as pictured in Figure 2. Moisture, ash content and volatile matter decreases as activation time increases. Meanwhile fixed carbon increases as activation time increases. The longer the reaction time, more moisture and volatile matter exit the activated carbon and produce a lower moisture and volatile matter while increase fixed carbon level.
Based on carbonization result, we acknowledge that the highest iodine number was produced through 900°C temperature carbonization process, which is 1150.1 mg/g. Based on that, activation process was conducted through 900°C temperature with time variation of 60, 90, and 120 minutes. As shown in Table 3, we can see that the largest iodine number was produced through 900°C activation temperature for 120 minutes that produce value of 1274.8 mg/g. This result is shown because in 120 minutes time, there are more volatile matter exits the sample, which produced more micro pores.

| Parameter          | 60 Minutes Thermal Activation | 90 Minutes Thermal Activation | 120 Minutes Thermal Activation |
|--------------------|-------------------------------|-------------------------------|-------------------------------|
| Yield%             | 53                            | 51                            | 49                            |
| Iodine number, mg/g| 1181                          | 1213.9                        | 1274.8                        |

As shown in Table 4, we can see that the activated carbon produced possesses more iodine number than the comparing activated carbon, which are Norit and Charcoal, Ajax Chemical. Based on that, we can conclude that low rank coal such as lignite possesses a great potential as activated carbon production main material with its high absorption capacity.

| Activated carbon                  | Iodine Number (mg/g) |
|-----------------------------------|-----------------------|
| Trademark: X                      | 828.7                 |
| Trademark: Y                      | 848.9                 |
| Experiment Resulted Activated Carbon | 1274.8               |

**4. Conclusion**

Activated carbon of low rank coal lignite was successfully synthesized through carbonization and activation phases with temperature variation of 400°C – 900°C and time variation of 60, 90, and 120 minutes. The largest iodine number was produced through activation with 900°C temperature for 2 hours, which is 1274.8 mg/g. The Synthesized resulted activated carbon iodine number is higher than comparing activated carbon. This shows that low rank coal such as lignite can act as activated carbon precursor with its high absorption level.
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