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# Preparation of Bagasse-Based Granular Activated Carbon by Chemical Activation Using Physical Mixing Method for Adsorbed Natural Gas

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## ABSTRACT

The use of activated carbon as adsorbent for adsorbed natural gas (ANG) gives the possibility of using agricultural waste as precursor. A study invented extremely high specific surface area of bagasse-based activated carbon through dry chemical activation. This experiment tried to get the optimum adsorbent for commercial ANG, wherein specific surface area, particle and pores size were considered. The steps of experiment were started by drying at 105 °C for 30 minutes, carbonization at 350 °C for 1-hour, granular size screening, chemical activation by physical mixing method with carbon and KOH ratio of 1:1 in the presence of $N_2$ of 500 ml/min at 700 °C for 1-hour, and then neutralization. Characterization gave $S_{BET}$ of 1576.40 $m^2/g$, average pore width of 2.37 nm, Scanning Electron Microscopy image, and elemental composition as identified by Energy Dispersive X-Ray Spectroscopy. These results demonstrate the potential of this product as an ANG adsorbent.

Keywords: activated carbon, adsorbed natural gas, chemical activation, sugarcane bagasse.

## 1. Introduction

Current natural gas storage and transportation systems are in the form of Compressed Natural Gas (CNG) and Liquefied Natural Gas (LNG) that involves high-cost process, specialized storage tank and pose safety risk due to the use of high pressure and/or cryogenic process. A promising alternative that will reduce cost and safety risk is the low-cost storage of natural gas at low pressure and normal temperature in the form of Adsorbed Natural Gas (ANG).

Activated carbon possesses advantages that make it suitable for gas storage such as high surface area, large pore volume, light weight and low cost. Another advantage of using activated carbon as an adsorbent is the possibility of using organic materials rich in carbon content as precursors. Besides the use of coal and wood, there is an increasing interest in the production of activated carbon from agricultural waste. The use of this material can reduce pressure on mines and forests while utilizing waste that has a good impact on the economy and the environment [1].

Bagasse-based activated carbon with a specific surface area of 3,554 $m^2/g$ has been successfully synthesized by a dry chemical activation process using KOH powder with a ratio of bagasse carbon: KOH of 1:4 at 800 °C [2]. The carbon content of bagasse is quite high, that is 24.7% [3]. The high cellulose and low lignin content in bagasse were indicated to produce a micropore structure on activated carbon which can increase the ANG capacity [4]. The use of KOH as an activating agent to produce activated carbon with a water content of 13.6% and ash content of 9.4% obtained iodine number of 884 mg/g and a surface area of 1,115 $m^2/g$ [5]. The best of activated carbon is activated by KOH impregnation and physical activation at 750°C, which obtained iodine number of 612 mg/g [6].

Activated carbon preparation involves two main steps: pyrolysis or carbonization and activation. In carbonization, the pore development is better defined with increasing temperature. Thermal decomposition of hemicellulose occurs above 200 °C [7]. Cellulose is degraded at a temperature of 315 – 400 °C and lignin is degraded at a temperature of 160 – 900 °C [8]. *In situ* activation in the presence of KOH molten salt media designs microporous carbon with high specific surface areas [9]. A general reaction between carbon and KOH is presented in Eq. (1):

$$6KOH + 2C \rightarrow 2K + 3H_2 + 2K_2CO_3 \quad (1)$$

KOH is entirely consumed at ~600 °C. At temperatures higher than 700 °C, the as-formed $K_2CO_3$ starts to decompose into $CO_2$ and $K_2O$ and vanishes at 800 °C.

$$K_2CO_3 \rightarrow K_2O + CO_2 \quad (2)$$

The higher activation temperature leads to wider micropores. Consequently, there is an increase in the specific surface area and pore volume [9]. ANG system requires specific surface area of 1000 $m^2/g$ – 3000 $m^2/g$ with average micropore width of 2.0 nm [10, 11].

Granular size of carbon can reduce the mass lost in the activation and handling process which can happen if the particle size is extremely fine and light. Extremely fine particles can be carried away by the flow of $N_2$ during activation and makes handling difficult because it is easy to fly considering that bagasse particles are very light. A wider particle size distribution can increase the density of adsorbent. This resulted in a corresponding increase in the methane (natural gas) storage density. Increasing the packing density of an adsorbent with the correct pore size distribution is a more practical solution than increasing the activity level [12]. The granular particle size also can increase the desorption capacity. However, the pressure in the adsorption tank will be longer [13].

Based on the background described above, this research tried to obtain activated carbon with optimum methane storage capacity for ANG by processing bagasse with KOH as an activating agent through physical mixing method during chemical activation.

## 2. Material and Methods

Dry bagasse is from PT Kebon Agung Sugar Factory Unit Trangkil, Pati - Central Java, Indonesia. Technical KOH with 90% purity was used in the form of flakes that was ground into powder.

### *2.1. Drying and Carbonization*

Bagasse was dried at 105 °C in oven for 30 minutes to ensure there is no water remains. Amount of dry bagasse were weighed and carbonized in muffle furnace at 350 °C for 1 hour with temperature increment of 10 °C/min under minimum oxygen condition that was obtained by covering the porcelain crucible with aluminium foil and given several small holes for removing volatile matter. The carbon was then weighed to get the carbonization yield data [2, 8].

The carbon was sifted with 10 and 80 mesh sieves to equalize the size. Therefore the granular carbon was obtained between 10 mesh (1.68 mm) to 70 mesh (0.17 mm).

### *2.2. Activation and Neutralization*

Carbon and KOH were mixed in a ratio of 1:1 in dry condition. Weighing was conducted before and after mixing. Then sample was then activated in a tubular furnace at 700 °C for 1 hour with a flow of 500 ml/min $N_2$ [2]. Weighing of the sample was conducted to calculate the activation yield.

Sample were neutralized using 0.5 N HCl solution, then filtered and rinsed with distilled water for three times. The sample were then dried in an oven at 105°C for 2 hours. Weighing of the sample after neutralization and rinsing was conducted to obtain the activation total yield. This activated carbon refers to bagasse-based activated carbon (BAC) sample.

### *2.3. Characterization*

The characterizations conducted were the iodine number analysis that refers to ASTM D4607-94 Standard Test Method for Determination of Iodine Number of Activated Carbon [14], Brunauer-Emmett-Teller (BET), Energy Dispersive X-Ray Spectroscopy (EDS) and Scanning Electron Microscopy (SEM).

## 3. Results and Discussions

### *3.1. Bagasse Water Content*

The bagasse obtained from the sugar factory is already in the dry form. However, drying is still needed to ensure there is no water content. Drying time of 30 minutes and 150 minutes produced the same water content data, therefore 30 minutes drying is enough to evaporate the water contained. The drying yield and water content data is presented in Table 1. The yield of drying process is 91.11 %wt in average and the bagasse water content is 8.89 %wt in average.

Table 1. Drying Yield and Water Content of Bagasse

| Bagasse Sample | Before Drying (gram) | After Drying (gram) | Yield (%wt) | Water Content (%wt) |
|---|---|---|---|---|
| 1 | 115.08 | 107.44 | 93.36 | 6.64 |
| 2 | 65.14 | 58.59 | 89.94 | 10.06 |
| 3 | 65.17 | 54.07 | 82.97 | 17.03 |
| 4 | 129.23 | 119.75 | 92.66 | 7.34 |
| 5 | 440.22 | 396.74 | 90.12 | 9.88 |
| 6 | 340.37 | 309.29 | 90.87 | 9.13 |
| 7 | 384.95 | 360.57 | 93.67 | 6.33 |
| 8 | 233.45 | 219.84 | 94.17 | 5.83 |
| 9 | 100.78 | 93.68 | 92.95 | 7.05 |
| 10 | 206.49 | 186.27 | 90.21 | 9.79 |
| 11 | 282.83 | 258.27 | 91.32 | 8.68 |
| Total | 2363.71 | 2164.51 | | |
| Average | | | 91.11 | 8.89 |

### *3.2. Carbonization*

Carbonization is the process of carbon formation and removal of volatile matter identified by the appearance of smoke. Carbonization is the process by which a carbonaceous material such as biomass is heated in the absence of air (oxygen) to form a porous solid. Carbonization produces a stable structure, with a partially developed pore structure [15].

Thermal decomposition of hemicellulose occurs above 200°C; specifically, in the temperature range of 250°C–400°C with release of CO and $CO_2$ from the *glycopyranose* ring [7], with the fastest decomposition at about 270°C. Cellulose is degraded at a temperature of 315°C–400°C with the fastest decomposition at a temperature of around 350°C. Lignin is slowly degraded at a temperature range of 160°C – 900°C [8].

In the process of sugarcane bagasse carbonization, mass loss occurs when the temperature is increased. The largest mass loss occurred in the cellulose content and the least loss in lignin. The resulting bagasse carbon has a brittle structure compared to bagasse, therefore easy to adjust the particle size as needed.

The carbonization yield data is presented in Table 2. The mass loss in carbonization process is 50.10 %wt in average. Meanwhile, the carbonization yield is 49.90 %wt in average. This yield is higher than the bagasse that was carbonized at 500°C with a yield of 25 %wt [2].

Table 2. Carbonization Yield of Bagasse

| Bagasse Sample | Dry Bagasse (gram) | Carbon (gram) | Mass Loss | | Yield (%wt) |
|---|---|---|---|---|---|
| | | | (gram) | (%wt) | |
| 1 | 24.00 | 11.40 | 12.60 | 52.50 | 47.50 |
| 2 | 26.30 | 12.60 | 13.70 | 52.09 | 47.91 |
| 3 | 24.80 | 11.80 | 13.00 | 52.42 | 47.58 |
| 4 | 15.70 | 8.20 | 7.50 | 47.77 | 52.23 |
| 5 | 19.70 | 12.70 | 7.00 | 35.53 | 64.47 |
| 6 | 9.50 | 4.20 | 5.30 | 55.79 | 44.21 |
| 7 | 5.80 | 3.30 | 2.50 | 43.10 | 56.90 |
| 8 | 13.30 | 5.90 | 7.40 | 55.64 | 44.36 |
| 9 | 15.50 | 6.90 | 8.60 | 55.48 | 44.52 |
| 10 | 13.60 | 6.10 | 7.50 | 55.15 | 44.85 |
| 11 | 13.60 | 6.10 | 7.50 | 55.15 | 44.85 |
| 12 | 18.20 | 9.80 | 8.40 | 46.15 | 53.85 |
| 13 | 11.00 | 4.20 | 6.80 | 61.82 | 38.18 |
| 14 | 7.00 | 3.40 | 3.60 | 51.43 | 48.57 |
| 15 | 14.20 | 7.40 | 6.80 | 47.89 | 52.11 |
| 16 | 12.80 | 6.70 | 6.10 | 47.66 | 52.34 |
| 17 | 12.90 | 6.70 | 6.20 | 48.06 | 51.94 |
| 18 | 17.00 | 8.80 | 8.20 | 48.24 | 51.76 |
| 19 | 19.50 | 12.20 | 7.30 | 37.44 | 62.56 |
| 20 | 13.50 | 5.50 | 8.00 | 59.26 | 40.74 |
| 21 | 6.60 | 3.70 | 2.90 | 43.94 | 56.06 |
| 22 | 15.20 | 8.00 | 7.20 | 47.37 | 52.63 |
| 23 | 15.50 | 8.10 | 7.40 | 47.74 | 52.26 |
| 24 | 14.70 | 7.70 | 7.00 | 47.62 | 52.38 |
| 25 | 15.40 | 8.20 | 7.20 | 46.75 | 53.25 |
| 26 | 15.90 | 10.30 | 5.60 | 35.22 | 64.78 |
| 27 | 12.50 | 5.00 | 7.50 | 60.00 | 40.00 |
| 28 | 7.60 | 4.30 | 3.30 | 43.42 | 56.58 |
| 29 | 14.40 | 7.20 | 7.20 | 50.00 | 50.00 |
| 30 | 14.30 | 7.30 | 7.00 | 48.95 | 51.05 |
| 31 | 16.20 | 8.10 | 8.10 | 50.00 | 50.00 |
| 32 | 15.50 | 7.80 | 7.70 | 49.68 | 50.32 |
| 33 | 18.80 | 11.90 | 6.90 | 36.70 | 63.30 |
| 34 | 12.70 | 5.20 | 7.50 | 59.06 | 40.94 |
| 35 | 7.80 | 4.40 | 3.40 | 43.59 | 56.41 |
| 36 | 15.10 | 6.60 | 8.50 | 56.29 | 43.71 |
| 37 | 14.40 | 6.30 | 8.10 | 56.25 | 43.75 |
| 38 | 13.90 | 6.00 | 7.90 | 56.83 | 43.17 |
| 39 | 13.60 | 5.90 | 7.70 | 56.62 | 43.38 |
| 40 | 10.10 | 4.90 | 5.20 | 51.49 | 48.51 |
| 41 | 14.10 | 6.90 | 7.20 | 51.06 | 48.94 |
| 42 | 13.80 | 6.80 | 7.00 | 50.72 | 49.28 |
| 43 | 14.00 | 6.80 | 7.20 | 51.43 | 48.57 |
| 44 | 13.20 | 6.40 | 6.80 | 51.52 | 48.48 |
| 45 | 10.90 | 6.90 | 4.00 | 36.70 | 63.30 |
| 46 | 6.70 | 3.70 | 3.00 | 44.78 | 55.22 |
| 47 | 15.40 | 7.00 | 8.40 | 54.55 | 45.45 |
| 48 | 14.30 | 6.60 | 7.70 | 53.85 | 46.15 |
| 49 | 14.00 | 6.50 | 7.50 | 53.57 | 46.43 |
| 50 | 15.20 | 6.90 | 8.30 | 54.61 | 45.39 |
| 51 | 12.20 | 6.70 | 5.50 | 45.08 | 54.92 |
| 52 | 7.40 | 3.70 | 3.70 | 50.00 | 50.00 |
| 53 | 12.40 | 5.70 | 6.70 | 54.03 | 45.97 |
| 54 | 15.90 | 7.40 | 8.50 | 53.46 | 46.54 |
| 55 | 15.20 | 7.00 | 8.20 | 53.95 | 46.05 |
| 56 | 10.50 | 5.60 | 4.90 | 46.67 | 53.33 |
| 57 | 6.50 | 3.00 | 3.50 | 53.85 | 46.15 |
| Total | 789.80 | 394.40 | 395.40 | | |
| Average | | | | 50.10 | 49.90 |

### *3.3. Chemical Activation*

The higher activation temperature leads to wider micropores. At high ratio of KOH, the micropores enlarge to become mesoporous, due to excessive KOH [9]. Meanwhile, the ANG requires the narrow micropore with a pore size distribution ranging from 1.0 to 2.0 nm [10]. However, higher activation temperature and high ratio of KOH could decrease the activated carbon yield as presented in Eq. (3) until Eq. (5). High KOH ratio also could harm the equipment as result of its corrosive property.

$$CO_2 + C \rightarrow 2CO \quad (3)$$

$$K_2CO_3 + 2C \rightarrow 2K + 3CO \quad (4)$$

$$C + K_2O \rightarrow 2K + CO \quad (5)$$

Purified nitrogen is an inert gas that can decrease the burn off precursor or formation of ash during pyrolysis process. The nitrogen may also function to accelerate the evaporation of unwanted materials. Beside that it may be able to support the formation of a new pore for increasing the surface area of activated carbon.

A general reaction between carbon and KOH is presented in Eq. (1). KOH is entirely consumed at ~600 °C. As a result of the operation temperature is 700 °C, the as-formed $K_2CO_3$ was not decomposed into $CO_2$ and $K_2O$. However, it reacts with HCl on neutralization process to produce potassium salt, $CO_2$ and water as presented in Eq. (6). The release of $CO_2$ was seen from the appearance of bubbles during neutralization. The potassium salt was dissolved in water and was separated from the activated carbon during rinsing.

$$K_2CO_3 + HCl \rightarrow H_2O + CO_2 + KCl \qquad (6)$$

The yield of activation before neutralization is 71.99 %wt in average and yield for neutralization process is 37.04 %wt in average. The total yield of activation process is 55.90 %wt. This yield is lower than the bagasse-based carbon that was activated with the same method at 800°C and carbon:KOH of 1:2 with a yield of 78 %wt [2]. The lower ratio of KOH gave higher yield in this activation method. However, in this experiment, the cellulose and lignin had not been fully degraded at 350 °C in carbonization process yet. It was continued degraded at 700 °C in activation process until the yield reached the amount of fixed carbon contained.

The Rolled Throughput Yield (RTY) of BAC preparation in this experiment according to Eq. (7) and Eq. (8) is 25.41 %wt. This is in accordance with the fixed carbon of bagasse which is 20 – 30 %wt [3].

RTY = Pass Yield (1) x Pass Yield (2) x ... (7)

RTY = Drying Yield x Carbonization Yield x Activation Yield ………………… (8)

Table 3. Activation Yield of Bagasse-Based Activated Carbon

| Carbon Sample | Carbon (gram) | Activated Carbon (gram) | Mass Loss | | Yield (%wt) |
|---|---|---|---|---|---|
| | | | (gram) | (%wt) | |
| 1 | 20.00 | 10.74 | 9.26 | 46.30 | 53.70 |
| 2 | 20.06 | 11.17 | 8.89 | 44.32 | 55.68 |
| 3 | 20.02 | 11.53 | 8.49 | 42.41 | 57.59 |
| 4 | 20.07 | 12.00 | 8.07 | 40.21 | 59.79 |
| 5 | 20.02 | 10.40 | 9.62 | 48.05 | 51.95 |
| 6 | 20.02 | 11.73 | 8.29 | 41.41 | 58.59 |
| 7 | 20.03 | 11.60 | 8.43 | 42.09 | 57.91 |
| 8 | 20.03 | 11.63 | 8.40 | 41.94 | 58.06 |
| 9 | 20.18 | 11.46 | 8.72 | 43.21 | 56.79 |
| 10 | 20.15 | 10.43 | 9.72 | 48.24 | 51.76 |
| 11 | 20.09 | 11.14 | 8.95 | 44.55 | 55.45 |
| 12 | 20.11 | 10.76 | 9.35 | 46.49 | 53.51 |
| Total | 240.78 | 134.59 | 106.19 | | |
| Rata-rata (%wt) | | | | 44.10 | 55.90 |

### *3.4. Characterization*

The iodine number analysis that refers to ASTM D4607-94 gave iodine number of BAC of 1584.99 mg/g and $S_{BET}$ of 1576.40 $m^2/g$. It complies to the ANG requirement of specific surface area that is 1000 $m^2/g$ – 3000 $m^2/g$ [10].

Brunauer-Emmett-Teller (BET) analysis gave average pore width of 2.37 nm that described the BAC contains micropore and mesopore. These average pore width is close to the ideal activated carbon pore size for the ANG system that should have an average micropore width of 2.0 nm [16]. Adsorption in the mesopore of activated carbon could initiate second and third layer of methane adsorbed and could decrease the retention of methane to be desorbed [17].

Energy Dispersive X-Ray Spectroscopy (EDS) gave elemental composition of bagasse-based carbon as presented in Figure 1 and Table 4. Elemental composition of BAC is presented in Figure 2 and Table 5.

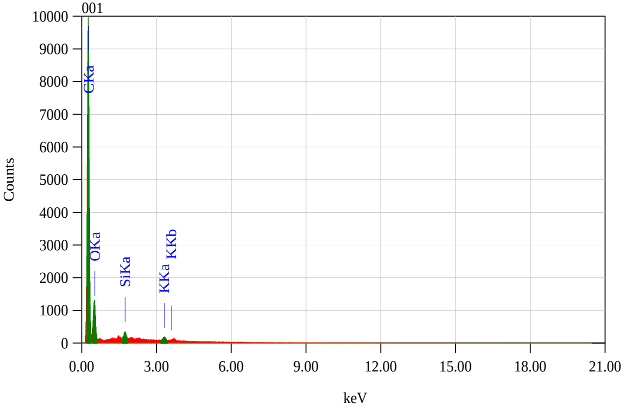

Figure 1. Elemental Composition of Bagasse-Based Carbon (EDS Result)

Table 4. Elemental Composition of Bagasse-Based Carbon (EDS Result)

| Element | keV | Mass % | Error % | Atom % | K |
|---|---|---|---|---|---|
| C K | 0.277 | 72.07 | 0.32 | 77.85 | 82.7505 |
| O K | 0.525 | 26.70 | 2.73 | 21.65 | 15.2195 |
| Si K | 1.739 | 0.66 | 0.61 | 0.31 | 0.9963 |
| K K | 3.312 | 0.56 | 0.77 | 0.19 | 1.0337 |
| Total |  | 100.00 |  | 100.00 |  |

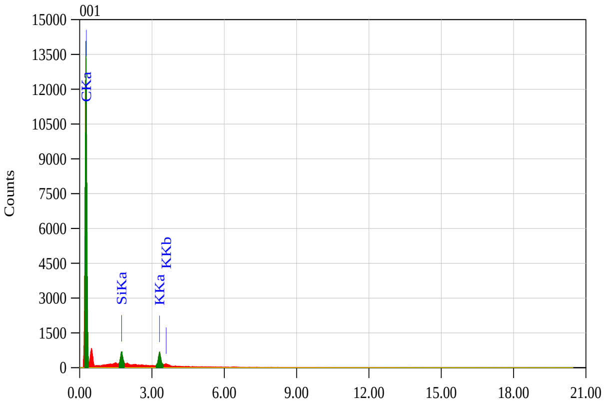

Figure 2. Elemental Composition of BAC (EDS Result)

Table 5. Elemental Composition of BAC (EDS Result)

| Element | keV | Mass % | Error % | Atom % | K |
|---|---|---|---|---|---|
| C K | 0.277 | 94.87 | 0.43 | 98.16 | 93.4316 |
| Si K | 1.739 | 1.66 | 0.91 | 0.73 | 1.9336 |
| K K | 3.312 | 3.47 | 1.24 | 1.10 | 4.6349 |
| Total |  | 100.00 |  | 100.00 |  |

EDS results show the carbon content is quite high in bagasse-based carbon and become higher in bagasse-based activated carbon (BAC), as the result of volatile matter that was completely released in higher temperature without significant loss of mass and ash formation. However, the kalium (K) element increases in BAC since the chemical activation use KOH as activating agent.

Scanning Electron Microscopy (SEM) produces detailed, magnified images of an object by scanning its surface to create a high-resolution image. The resulting images show information about its physical features. The physical features of bagasse-based carbon and BAC are presented in Figure 3 and Figure 4.

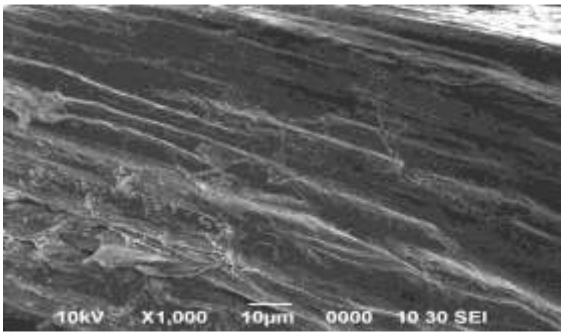

Figure 3. Physical Features of Bagasse-Based Carbon (1000x Magnification)

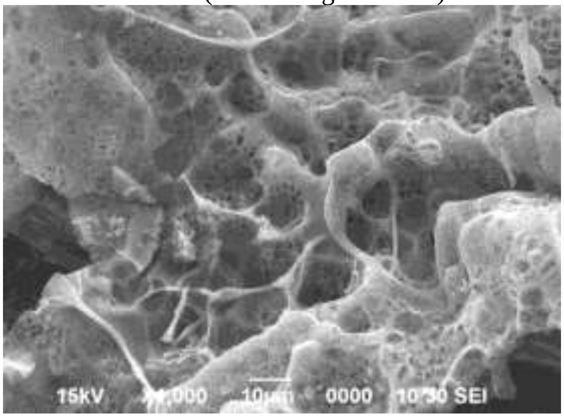

Figure 4. Physical Features of BAC (1000x Magnification)

SEM results show great amount of micropores and mesopores were well formed in the BAC which indicates that the activation process worked well.

## 4. Conclusion

The physical mixing method in dry condition between carbon and KOH in chemical activation process led to the formation of great amount of micropores and mesopores that result in high specific surface area.

Granular bagasse-based activated carbon with particle size distribution of 10 mesh (1.68 mm) to 70 mesh (0.17 mm) which can provide the advantages of easy handling and increasing the adsorbent density can have the specific surface area of 1576.40 $m^2/g$ by chemical activation process using physical mixing method between KOH and carbon (ratio of 1:1) at 700°C with $N_2$ flow of 500 ml/min. Therefore, it will allow high adsorption capacity of methane in ANG that requires specific surface area of 1000 $m^2/g$ – 3000 $m^2/g$.

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