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POTENTIAL STUDY OF INDONESIA COAL FOR ADSORBED NATURAL GAS

STUDI POTENSI BATUBARA INDONESIA UNTUK ADSORBED NATURAL GAS

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ABSTRACT

Low rank coal was used as a precursor for the preparation of adsorbed natural gas by chemical activation with KOH-NaOH mixtures. Chemical activation process is commonly used and involves two major steps, which are heating process and chemical treatment process. The experiments were used in the various of temperatures, contact times, and rasio of sample and chemical compound. The result of process was obtained of the iodine number around of 1004 mg/g and 1198 mg/g . Based on the other study, the iodine number at 1004 mg/g and 1198 mg/g have surface area between 1000 mg/g up to 1200 mg/g. These surface area provide a methane capacity between 75 mg/g up to 80 mg/g.

Keywords: activated carbon, adsorbed natural gas, surface area, iodine number.

ABSTRAK

Batubara peringkat rendah dapat digunakan sebagai bahan baku untuk pembuatan adsorbed natural gas dengan aktivasi kimia menggunakan campuran KOH-NaOH. Proses aktivasi kimia umum digunakan dan meliputi dua tahap utama, yaitu proses pemanasan dan proses perlakuan kimia. Percobaan dilakukan dalam berbagai temperatur, waktu kontak, dan rasio sampel dan campuran bahan kimia. Hasil proses dengan menggunakan dua jenis sampel batubara diperoleh nilai bilangan iodin sebesar 1004 mg/g and 1198 mg/g. Berdasarkan kajian literatur lain, bilangan iodin 1004 mg/g and 1198 mg/g memiliki luas permukan antara 1000 m^2/g sampai 1200 m^2/g. Surface area ini menyediakan kapasitas metana antara 75 mg/g sampai 80 mg/g.

Kata kunci: karbon aktif, gas alam teradsorpsi, area permukaan, nomor yodium

INTRODUCTION

Most of Indonesia's coal reserve is categorized as low rank coal. This type of coal should be prioritized for electricity (Sihite, 2012). In order to support the Indonesia Goverment Regulation No. 23 Year 2010 on Implementation of Activity Mineral and Coal Mining Enterprises, coal processing technology becomes more important and need to be developed in Indonesia. One of the coal processing technology currently be developed was activated carbon making from low rank coal (Hudaya, Sulistyohadi and Monika, 2014). Production of activated carbons (AC) is based mainly on the natural organic substrates, mainly hard coal, brown coal, wood, sawdust, local waste products, for example nut shells or fruit stones (fruits seeds). An attractive but much more expensive precursor of ACs are synthetic polymers, e.g. waste phenolic resins, phenol-formaldehyde resins or urea-formaldehyde resins, polyurethane and ethylene polyterephthalate (Nowicki, Kazmierczak and Pietrzak, 2015). Because of their unique porous structure, easy availability and neutral effect on the natural environment, ACs have found a wide range of applications.

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On the AC aplications is well known as one of the best adsorbents for gases (Cheng, Yang and Liu, 2001). The phenomenon of physical adsorption is essentially accumulation of the undissociated hydrogen molecules on a surface of microporous carbon fibers or particles. This property is due to the fact that the carbon could be prepared in a very fine powdered or fiber form with highly developed porous structure and due to specific nature of the interactions between carbon atoms and gas molecules (Vasiliev *et al.*, 2006). One of the AC aplications is used to Adsorbed Natural Gas.

There are three options available for selection in storing natural gases - liquefied natural gas (LNG), compressed natural gas (CNG) and adsorbed natural gas (ANG). In consideration of safety in the home environment, the LNG, which requires a low temperature, and the CNG, which requires a high pressure can be excluded from the list. The adsorbed natural gas (ANG) shows its potential as a way for storing natural gas because of its higher energy density under a much lower pressure than CNG (Gao, 2013). Using adsorbing porous solid support filling a new storage system seems to be an alternative technology to store hydrocarbons gas under convenient conditions for mobile uses (Yahia and Ouederni, 2012). Adsorbed Natural Gas (ANG) storage offers several advantages over Compressed Natural Gas (CNG) or Liquefied Natural Gas (LNG) storage. In ANG system natural gas is stored in the adsorbed phase using some highly micro porous adsorbent like activated carbon (Sahoo and Ramgopal, 2014). However, unlike CNG or LNG, the performance of ANG systems depends on a large extent on the heat and mass transfer aspects of the storage beds. Hence a large number of theoretical and experimental studies are carried out on heat and mass transfer aspects of ANG reactors in the past (Loh *et al.*, 2010). The research mainly focuses on searching for high performance adsorbents by way of management of thermal effect from adsorption and desorption. Adsorbents used are the highly micro-porous activated carbons with relatively high packing densities and specific surface area (Biloé, Goetz and Guillot, 2002).

Recently many improvements have been accomplished to obtain microporous carbonaceous materials with very high adsorbing properties. The study indicated that the properties of AC depend on the source and preparation conditions (Abechi *et al.*, 2013). There are two main processes for preparation of AC; chemical activation and physical activation. Chemical activation is known as a single step method of preparation of AC in the presence of chemical agents. The chemical activation usually takes place at a temperature lower than that used in physical activation, therefore it can improve the pore development in the carbon structure because of the effect of chemicals. The carbon yields of chemical activation are higher than physical activation (Sudaryanto *et al.*, 2006). Chemical activation process is commonly used and involves two major steps, which are heating process and chemical treatment process. The heating process usually requires less heat than the physical activation process. During chemical treatment, chemical agents such as alkaline chemicals (KOH, NaOH, Na_2CO_3 and $NaHCO_3$), acid chemicals (HCl, H_2SO_4 and $C_2H_4O_2$) and other chemicals ($ZnCl_2$; etc) are added to improve the surface area or size of porous structure in AC materials. Chemical activation processes are more preferred compared to physical activation due to the higher global yield (based on weight of starting material), simplicity, lower temperature and shorter activation time (Pragya, Sripal and Maheshkumar, 2013). Physical activation involves carbonization of a carbonaceous materials followed by activation of the resulting char. The physical methods are typically thermal processes conducted at temperatures below 700°C using oxidizing gases like air, CO_2 and steam of H_2O as second step after pyrolysis (Sekirifa *et al.*, 2013). This process is used to create a porous structure that increases the adsorption capacity of AC materials. The porous AC is produced when the oxidant converts carbon materials to form CO and CO_2 opening pores in AC materials (Ahmida *et al.*, 2015). Physical and chemical characteristics of AC affect its suitability for specific applications. Therefore, these processes need to be well studied in order to understand the relationship between applications and their properties (Ahmed and Theydan, 2012).

Although there are many studies in the literature relating to the preparation and characterization of AC from coal as mentioned before, there is no information for the preparation of AC using Indonesia coal for ANG as the precursor with mixture of KOH-NaOH as chemical activating agent. Therefore, these reseach is conducted as initial study to produce AC with the high surface area. In the present study, we used a bituminous and sub-bituminous coal as material for AC. The main objective in this work is to study the effect of different preparation variables on the adsorption properties of activated products for ANG.

EXPERIMENTAL

Preparation of AC

AC was prepared using two samples of sub-bituminous coal, which are sub-bituminous coal type A (SCTA sample) and sub bituminous type C (SCTC sample). Coal characteristic of the samples were showed in Table 1. Prior to the use, coal was crushed and sieved to particle size of -1 mm. In the first step, the preparation is conducted on SCTA. Coal was carbonized in a horizontal furnace equipped with temperature control (Figure 1).

Figure 1. Horizontal furnace

Carbonization process of SCTA sample was conducted at selected temperatures of 400, 450 and 500°C for each carbonization at 30, 60, and 90 minutes. Charcoal of carbonization were then analyzed to determine of fixed carbon and ash contents. The optimal conditions is indicated by highest content of fixed carbon and lowest content of ash. The optimal conditions of SCTA process was obtained will be used to the carbonization process of SCTC sample.

In the second steps, the charcoal of SCTA sample was activated using chemical compounds. In the activation process, charcoal was mixed with the 30% of KOH-NaOH mixture in a beaker glass. KOH : NaOH mixture was prepared with a mol ratio of 1:1. While ratio of charcoal : KOH : NaOH mixture was difined of 1 : 1, 1:3, and 1:4. After completion, SCTA sample was heated at 150-200°C for 1 hour up to almost dried. During the heating the mixtures of sample and KOH-NaOH were stirred automatically. The resulting SCTA sample was placed in the horizontal furnace and then re-heated at desired temperature of 800°C for 30, 60 and 90 minutes. The activation was performed under Nitrogen condition at a flow rate of 50 ml/min by heating rate of 5°C/min from room temperature (around 30°C) up to the desired temperature was reached. At the end of activation process, the SCTA was cooled at room temperature, for overnight. The SCTA sample was neutralized for several times with cold distilled water and sample was dried at 110°C for 2 hrs and stored in closed bottles. Finally, on SCTA sample was conducted iodine numbers analysis. On the SCTC sample, carbonization process was conducted at the optimal conditions that was resulted from the SCTA carbonization. While activation process was conducted at temperature of 750, 800, and 850°C. On each activation process residence times were varied of 30, 60 and 90 minutes.

Porous materials such as AC are usually characterized by several physical parameters such as surface area and pore volume. The surface area of activated carbon are usually measured using the Brunauer-Emment-Teller (BET) method, which employs the nitrogen adsorption at different pressure at the temperature of liquid nitrogen (Nunes and Guerreiro, 2011). Additional information about the structure of activated carbons can be obtained by the adsorption characteristics of differerent adsorbates, such as methylene blue and iodine adsorption. Adsorption experiments of this molecules are easy and habitually to characterize activated carbon with the purpose obtaining information on the adsorption capacity of the materials. According to the dimensions of the methylene blue molecule, it is mainly adsorbed in mesopores, however, a small portion is also found in larger micropores. In relation to methylene blue, the iodine molecule possesses inferior dimensions which make it penetration in micropores possible (Momen *et al.*, 2009). These characteristics confer such molecules the potential for using them as probes in the study of the physical structure AC.

In spite of this, no detailed study has been found concerning the quantitative realationships between methylene blue and iodinen numbers and the stuctural characteristics of the AC (Nunes and Guerreiro, 2011).

Activated Carbon Characterization

The characterization of activated carbon mainly through iodine number. The iodine number of AC indicates the ability of the AC to be an adsorbent. The iodine number was determined according to the ASTM D 4607-94 Standard Test Method for Determination of Iodine Numbers of Activated Carbon. The iodine number is defined as the milligrams of iodine adsorbed by 1,0 g of carbon when the iodine concentration of the filtrate is 0,02 N (0,02 mol/L). This method is based upon a three-point isotherm. A standard iodine solution is treated with three different weights of AC under specified conditions. The experiment consists of treating the AC sample with 10,0 ml of HCl 5%. This mixture is boiled for 30 s and then cooled. Soon afterwards, 100,0 ml of 0,1 N iodines solution is added to the mixture and stirred for 30 s. The resulting solution is filtered and 50,0 ml of the filtrate is titrated with 0,1 N sodium thiosulfate, using starch as indicator. The iodine ammount adsorbed per gram of carbon is plotted against the iodine concentration in the filtrate, using logarithmics axes. If the residual iodine concentration is not within the range of 0,008 to 0,04 N, the whole procedure should be repeated using different carbon masses for each isotherm point. A least squares fitting regression is applied for each isotherm point. The iodine number is *X/M* value when the residual concentration is 0,02 N. The *X/M* and *C* values are calculated by the below equations.To calculate the value of *X/M*, first derive the following values :

$A = (N_2)(12693,0)$
$B = (N_1)(126,93)$
$DF = (1 + H)/F$

where :
A = constanta value for iodine
B = constanta value for thiosulfate
N_2 = Normality of iodine
N_1 = Normality of thiosulfate
DF = dilution factor,
I = iodine, ml
H = 5% hydrochloric acid used, ml
F = filtrate, ml

For example, if 10 ml of HCl and 50 ml of filtrate are used :
DF = (100+10)/50 = 2,2

Calculate the value of X/M as follows.

$$X/M = [A - (DF)(B)(S)] / M$$

where ;
X/M = iodine absorbed per gram of carbon, mg/g
S = sodium thiosulfate, ml., and
M = mass of AC used, g

Calculate the value of C as follows.

$$C = (N_1 - S) / F$$

Where :
C = residual fltrate, N

RESULTS AND DISCUSSION

Coal Characterization

The proximate, ultimate and caloric value analysis of coal used in this study, are given in Table 1.

The elemental composition of the starting coals were determined on a proximate analysis according to the ASTM Standard Method. In four parameters in Table 1, the ash content is the important factor because to affect the process of porosity formation, also the utilization of AC. The second factor to affect is moisture content. The moisture content of coal is high, the acquisition of product is low. while on the carbonization process, volatile matter content will be drop and fixed carbon content will be rise. The results of analysis showed that ash content of less than 6,00% can produce a good adsorption capacity (Monika and Suprapto, 2009).

Table 1. Proximate and ultimate analysis of sub-bituminous coal

Analysis	SCTA (%)	SCTC (%)
Proximate		
Moisture	16.59	17.10
Volatile matter	39.08	39.98
Fixed carbon	42.60	37.96
Ash	1.73	4.96
Ultimate		
Carbon	63.51	47.94
Nitrogen	0.90	0.68
Hydrogen	6.62	4.75
Oxygen	27.03	28.30
Sulphur	0.21	0.66
	(Cal/g)	(Cal/g)
Calorie value	5973	4378

Charcoal Characterization

As mentioned above, the first stage of AC preparation was carbonization process. In this process, pyrolysis occured . Pyrolysis is a common stage of many coal utilization processes such as carbonization, gasification, combustion, liquefaction, which are operated usually in close systems under inert condition, reduction or oxidation atmospheres at different pressure and residence time. The products of the pyrolysis of coals are charcoal (or coke), liquids and gases (Rađenoviæ, 2006). However, generally in the manufacture of activated carbon is known as carbonization process. Furthermore, the chemical activation process is conducted on charcoal.

Table 2 shows charcoal characteristic for SCTA was prepared with different temperatures and residence times. One of the most important characteristics for activated carbon is ash content. The feed coal must have low ash content, since the percentage of mineral matter in the course of activaton is increased and the adsorption capacity decreased (Jüntgen, Knoblauch and Zündorf, 1973). The characteristics of SCTA was evaluated by proximate analysis. The different temperature and residence time were used to study the effect of carbonization process on moisture content, ash content, volatile matter content and fixed carbon content. Generally, ash content increased with increasing temperature of carbonization, while volatile matter content was decreased. The higher of fixed carbon content was obtained at carbonization temperature of 500°C for 30 minutes. While the lowest ash content with a carbon content of 69,40% is achieved with a residence time of 60 minutes. Therefore, the optimal conditions of the carbonization process is determined at a temperature of 500°C for 60 minutes.

This condition is used for the carbonization process of SCTC sample. The results of the carbonization process of SCTC sample are shown in Table 3. After carbonization, coal ash content increased from 4,96% to 7,54%. While fixed carbon content is low. This condition is caused a decrease of volatile matter content is small, from 39,98% to 30,62%. Different of SCTA sampel, a temperature of 500°C for 60 minutes can reduce volatile matter content to be less than 27%. The effect of low fixed carbon content is a small of percentage yield of AC product. On this condition, a temperature more than 500°C can increase of fixed carbon content of SCTC sample.

Iodine Number of AC

AC were prepared of SCTA sample in the presence of KOH-NaOH at 800°C for 90 minutes by activation of charcoal. All the AC products were studied by physical adsorption of iodine numbers. The highest iodine numbers indicated that AC has high capacity adsorption. It was found that a high adsorption capacity has a large surface area and microporous volume is high (Menon and Komarneni, 1998). Figure 2 shows iodine number of AC.

The curve of Figure 2 shown that a longer of residence time, the trend of iodine numbers decreased. In a residence time of 30 minutes, iodine numbers is stable. However, iodine numbers is increasing significantly at 60 minutes up to achieve of 1004 mg/g. At the same of residence time, effects of ratio 1:4 caused iodine numbers decreased significantly. In Figure 2 indicated that the highest of iodine numbers is reached at 60 minutes with a ratio of 1 : 3.

For SCTC sample, activation is conducted at different temperatures of 750, 800 and 850°C and residence times of 30, 60 and 90 minutes. While ratio of sample : KOH-NaOH mixture according to the optimal condition of SCTA activation was 1 : 3.

Table 2. Charcoal characteristics of SCTA sample

Temperature (°C)	Residence time (min)	Proximate analysis			
		Moisture (% adb)	Ash (% adb)	Volatile matter (% adb)	Fixed carbon (% adb)
450	30	3.18	2.30	32.37	62.14
	60	3.86	2.56	27.24	66.34
	90	3.58	3.02	27.05	66.35
500	30	1.61	3.06	23.03	72.29
	60	1.66	2.66	26.28	69.40
	90	1.24	3.12	25.09	70.55
600	30	3.44	5.,75	23.52	67.29
	60	2.42	6.98	22.04	68.56
	90	1.99	6.76	21.76	69.49

Table 3. Charcoal characteristics of SCTC sample

Sample name	Moisture (% adb)	Ash (% adb)	Volatile matter (% adb)	Fixed carbon (% adb)
SCTC charcoal	6.31	7.54	30.62	55.52

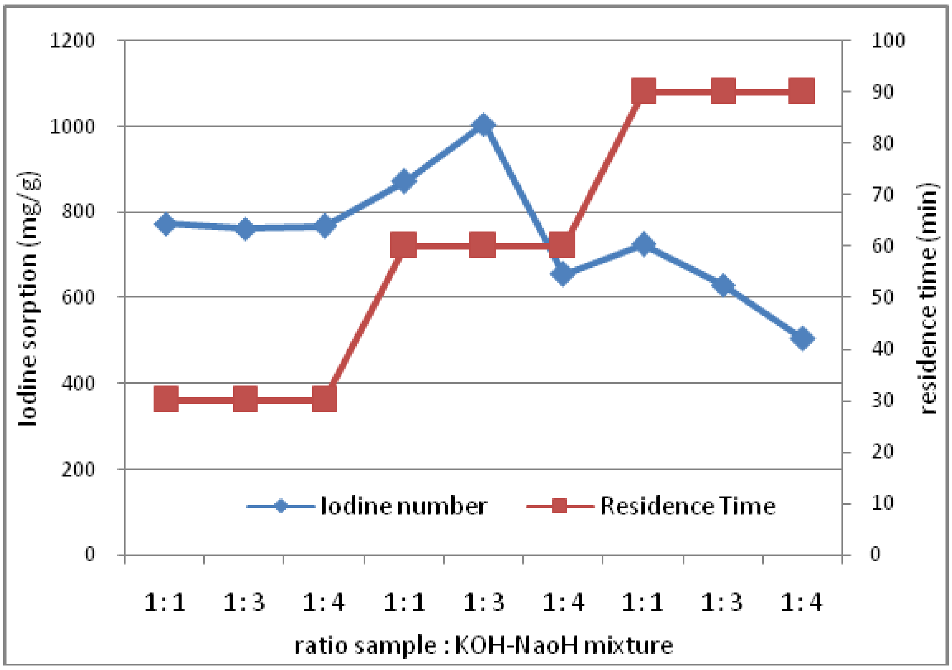

Figure 2. Iodine numbers of SCTA sample

As shown in Figure 3, at temperature of 750°C the additional of activation time from 30 minutes up to 60 minutes resulted in rise of iodine numbers. At activation time of 90 minutes, curves indicated in decline of iodine numbers. Similarly, at temperature of 800°C and activation time of 30 minutes up to 60 minutes, curves in Figure 2 shown in rise of iodine numbers. However at activation time of 90 minutes, iodine numbers signicantly declined up to less then 600 mg/g. The additional temperature up to 850°C shown that activation with ratio sample/KOH-NaOH mixture resulted in rise of iodine numbers. According to Wang and Gao, in their study in (1991), the activation of carbonized materials can be divided into two steps. The first is the selective forming of material to be amorphous carbon to form microporous structures through the opening of closed pores, the second is the successive forming of carbon crystallites to form mesoporous and/or macroporous structure through the breaking of walls between micropores.

Figure 2 and Figure 3 show that the KOH-NaOH mixture on prepared of activated carbon gave highest Iodine number value of 1004 mg/g (SCTA sample) and 1198 mg/g (SCTC sample). Iodine numbers is a measure of the micropore content of the activated carbon (0 to 20 Å, or up to 2 nm).

The higher Iodine number of the activated carbon has been attributed to the presence of large micropore structure might have resulted due to the reactivity of the activating agent KOH (Joshi and Pokharel, 2014). During carbonization and activation process, oxygen of the KOH could remove cross-linking and stabilizes the carbon atoms in crystallites. Removal of potassium salts from the internal volume of the carbon by washing creates the micro-porosity in the activated carbon (González *et al.*, 2009). In general the adsorption capacity of an adsorbent like AC is the function of surface area, pore volume, pore size distribution and surface chemistry. There are numerous reports of more organic adsorption in pores that are barely large enough to admit the adsorbate molecule (Jalil, 2012).

In adsorbed natural gas, the indication of gas adsorption was performed of methane adsorption. Adsorption amount of methane was directly related to surface area. The adsorbent for ANG has to be predominantly microporous. Optimal storage capacity will occur when that fraction of the storage volume that is micropore is maximized, with no void or macropore volume. However, this contribution has to come from pores with size greater than at least 7.6 °A (i.e., greater than the thickness of two methane molecules), in order to maximize the deliverability at ambient pressure. In addition to having a high surface area, low mesoporosity is desirable. The presence of low mesoporosity in the form of feeder pores with size less than 50A° is required to some extent in order to provide easy access for the adsorbate molecules to/from the micropores (Menon and Komarneni, 1998).

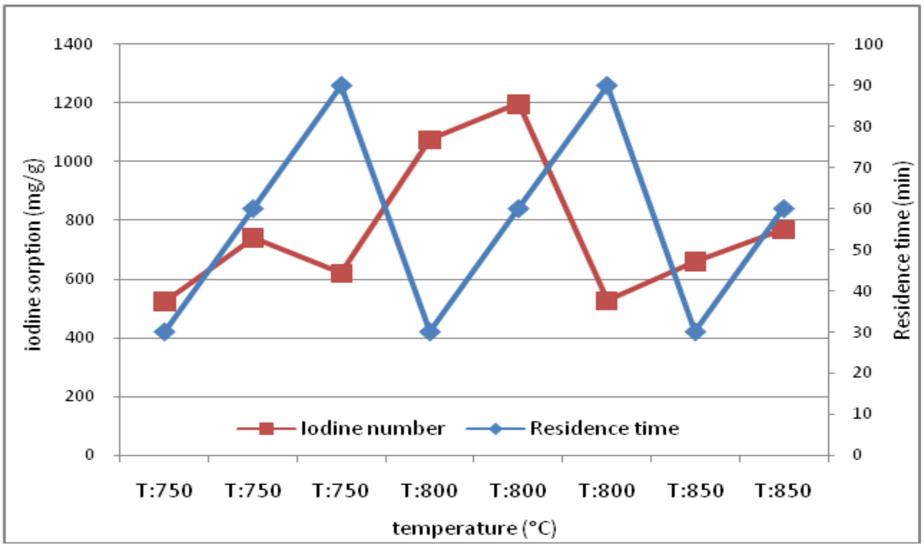

Figure 3. Iodine numbers of SCTC sample

Menon and Komarneni (1998), reported in their study that the higher of surface area, the greater the methane capacity. As listed in Table 4, the surface area of AC for ANG has the ability for methane gas adsorpsi in the various capacity.

Carbon	Surface area m^2/g	Methane capacity (mg/g)
Granular powder		
MSC-5A	445	44
Carbon Lorraine	640	75
Saran (B)	900	87
BPL	1030	75
Norit R1	1240	82
Amoxo GX-32	2500	175
Kansai coke based	3100	211
Norit WX6	445	48
Calgon SGL	900	65
AC carbon CNS96	1190	80
Norit R3	1270	92
Kareha BAC	1350	99
California GMS-70	1502	108
Barnebey MI	1730	109
Sutcliff GS 60	1860	129
Osaka M-30	2415	144
Kansai Maxsorb	2671	164
Electrosynthesis EL	2796	170

The surface area of 3100 m^2/g has the highest methane capacity at 211 mg/g, while the surface area of 445 m^2/g has the lowest methane capacity at 44 mg/g. In this study, to determine of methane capacity of coal AC was conducted by data estimation and comparing to data in Table 4. Based on the study of Code and Provenzano (2012), the iodine number at 1004 mg/g and 1198 mg/g have surface area between 1000 m^2/g up to 1200 m^2/g. According to data in Table 4, these surface area provide a methane capacity between 75 mg/g up to 80 mg/g.

The results of this study showed that Indonesia coal potensial to be prepared for ANG as storage of natural gas. The adsorption capacity is quite good. It was indicated by a methane capacity value was not lowest.

CONCLUSION

Activated carbon was prepared from Indonesia coal by chemical activation with KOH-NaOH mixture in ratio of 1:3 by weight at 800°C for 60 minutes. The prepared activated carbon was characterized by determining iodine number value. The results of study indicate that KOH-NaOH mixture can increase micropore volume and based of the literature has adsorption methane capacity between 75 mg/g up to 80 mg/g.

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